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RESEARCH ON THE
PHYSICAL PROPERTIES
OF MARINE SOILS

August 1961—July 1962

DEPARTMENT
OF
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MASSACHUSETTS INSTITUTE OF TECHNOLOGY
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Report for the Period
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Soil Engineering Division
Department of Civil Engineering
Massachusetts Institute of Technology

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ABSTRACT

This report describes in detail a method for quantitatively measuring fabric of clay.

Wet clay is impregnated with a polyalcohol which permits grinding a very flat surface (without disrupting the original fabric) for quantitative X-ray measurement. In the specific procedure discussed, a Norelco diffractometer equipped with a pole figure device and a Geiger detector was employed to measure by reflection the diffracted intensity of selected peaks (002 and 020) on kaolinite clay. The amplitude of the 002 peak to the amplitude of the 020 peak is called the peak ratio which quantitatively expresses particle orientation on a given surface. Heterogeneity of particle orientation on a surface is expressed in terms of the observed standard deviation relative to the standard deviation expected from counting statistics for peaks of the appropriate amplitude.

Utility of the method is shown by detailed data for isotropic and anisotropic consolidation and for a sheared sample. Data on engineering samples are compared to artificially defined ideal random and ideal orientation specimens of the same kaolinite. Isotropic consolidation of flocculated kaolinite slurry to 1 kg./cm² remains nearly as random as air-dry kaolinite powder (ideal random). Anisotropic (one-dimensional) consolidation of flocculated kaolinite slurry to 197 kg./cm² is nearly as well oriented as thoroughly dispersed kaolinite slurry dried onto a glass microscope slide (ideal orientation). Maximum orientation in the shear zone which is about 30° to the consolidation pressure, $\bar{\sigma}_c$, is at least as great as the orientation at 90° to $\bar{\sigma}_c$ well outside the shear zone. Orientation at 30° to $\bar{\sigma}_c$ outside the shear zone is 1/4 the maximum in the shear zone.

PREFACE

Since November 1952, the Office of Naval Research has sponsored research in the Soil Engineering Division of the Department of Civil Engineering of the Massachusetts Institute of Technology. For the past four years, the studies have been concerned with the influence of environmental changes on the engineering properties of clay soils. Emphasis was placed on determining the effects of geological changes on shear strength, especially on marine sediments. The geological changes included cation exchange, potassium fixation, and cementation by carbonates. For the past one and one-half years, a fundamental study of clay fabric has been carried out in order to develop a quantitative measure of the geometrical arrangement of clay particles. Such information is required for a realistic assessment of the fundamental nature of shear strength in clays and hence for an explanation of the effects of environmental changes on the engineering properties of clay soils.

This report, for the period August 1961 through July 1962, describes in detail the research on quantitative determinations of clay fabric. The work was performed by Dr. R. Torrence Martin, Research Associate, aided by Dr. Ewa Paszyc-Stepkawska, Research Associate, under the general supervision of Dr. T. William Lambe, Professor and Head of the Soil Engineering Division. Dr. Charles C. Ladd, Assistant Professor, helped in the role of technical advisor. The report was prepared by Dr. Martin.

Pertinent reports issued under this research contract during the past three years are:

1. "Research on the Physical Properties on Marine Soils," Soil Engr. Div. Publ. 105, M.I.T., July (1960).
2. "Research on the Physical Properties of Marine Soils," Soil Engr. Div. Publ. 117, M.I.T., July (1961).
3. "Research on the Fundamentals of Soil Behavior - Progress Report for September 1961 - February 1962," Soil Engr. Div. Publ. 122, Dept. of Civil Engr., Publ. R62-14, M.I.T., June (1962).

I. INTRODUCTION

Behavior of clay-water systems, over a wide water content range, has been explained by various authors (van Olphen, Lambe, Seed, Chan, Olsen, and others) in terms of the structure of the clay phase. Structure is the combination of the geometrical arrangement of particles and the forces operating between them. The geometrical arrangement of particles is called fabric and is the component of structure that is most amiable to measurement. Since the structure concept has been invoked to explain so many features of clay behavior, a reliable quantitative method for determining the fabric of a moist clay is extremely important.

The problem of measuring fabric is actually two inter-related problems: 1) to prepare a specimen for examination that preserves the fabric of the original sample, and 2) to examine some property of the clay crystallites that quantitatively expresses their geometrical arrangement. Procedures that have been employed to measure particle arrangement include light and electron microscopy, and X-ray diffraction. Specimen requirements are different for these various examination techniques; therefore, the details of sample preparation are controlled, in part, by the procedure to be used for examination. Neither light nor electron microscopy results are easily expressed quantitatively, although Kubiena, et al (1961) has obtained quantitative data on void sizes and distribution by light transmission measurements. Brindley (1953) demonstrated that the X-ray powder camera could be used to quantitatively measure fabric. A slight modification of Brindley's technique was employed by Raithburd (1958 and 1960) on dried clay specimens. While Raithburd's X-ray data are quantitative, the specimens were dried prior to preparation for X-ray which seriously limits interpretation of the X-ray data relative to the fabric of the

original wet clay. Mitchell (1956) made an innovation toward minimizing disruption of the original fabric by gradually replacing the water with a polyalcohol which is easily solidified to give the clay rigidity.

A detailed experimental procedure along with illustrative data on the fabric of a kaolinite clay are the subject of the present discussion. Briefly, the technique employed is: 1) to impregnate the wet clay with polyalcohol, 2) to grind a flat surface, and 3) to examine by X-ray diffraction using a diffractometer equipped with a Geiger detector for recording quantitative intensities on selected 00 ℓ and hko peaks of the clay mineral.

II. X-RAY DIFFRACTION

The principle behind the study of fabric by X-ray diffraction is very simple; however, satisfactory execution of the principle requires some care. The idea is to compare the relative diffracted intensity of a 00 l peak to that of an hko peak for the same mineral. This peak ratio (P.R.) is defined as the amplitude of the diffracted intensity for an 00 l peak divided by that for an hko peak.

An advantage of expressing the fabric or preferred orientation as a peak ratio is that the data are automatically corrected for variations in crystallite concentration in the specimen surface which may arise from either a void within the irradiated area or an overall low bulk density. On the other hand, different areas of a specimen surface may have different fabrics. The magnitude of this variation can be determined by obtaining the average peak ratio from a large area and the deviations from the average value by determining peak ratios for small areas within the large area. Therefore, the peak ratio provides a numerical value for the fabric of the surface examined as an average value and the deviation from that average.

The peak ratio for a perfectly oriented specimen would go from infinity for the plane parallel to 00 l to zero for the plane normal thereto. In order to establish the extreme peak ratio limits for any actual clay, severe laboratory pretreatments are employed. These pretreatments establish the extreme peak ratio limits which are designated as ideal random and ideal orientation. Comparison of the peak ratio, P.R., for an unknown fabric condition on a particular clay to the peak ratios for ideal random and ideal orientation on the same clay established the relative degree of preferred orientation for the unknown fabric.

Before one can determine the peak ratio change due to engineering manipulation of a clay, one has to locate the surface or zone of maximum fabric change relative to the original reference

plane. The pole figure attachment to the standard X-ray diffraction goniometer removes much of the guesswork necessary to locate the surface of maximum fabric change. Accordingly, a brief description of the pole figure device is given.* Constant reference to Fig. 1 will help to clarify the different specimen movements which are otherwise difficult to present clearly.

The standard diffractometer permits only one motion of the specimen, θ rotation. The center of the specimen face is on the axis of the goniometer and rotates about axis YY' . With the standard diffractometer, reflections can be recorded only from those crystallites which: 1) satisfy the Bragg equation and 2) are situated in a plane parallel to the axis of rotation, YY' .

The pole figure device permits three additional motions and at the same time maintains focusing geometry to satisfy the Bragg equation. These motions are: 1) tip, ϕ , the specimen rotates about the XX' axis; 2) spin, α , the specimen is rotated in its own plane (about the $00'$ axis); and 3) oscillation, osc , the specimen surface is translated in the XY plane along the YY' axis. Therefore, the pole figure device provides a means of examining the reflections from all surface crystallites.

It is necessary in the pole figure device to limit the width of the primary X-ray beam with a vertical slit in order to maintain correct diffraction geometry, especially for ϕ rotation. The vertical slit greatly reduces the intensity of the diffracted beam reaching the detector and the area of the specimen diffracting. By proper choice of power settings on the X-ray target and the width of vertical slits, one may examine a fairly narrow width of specimen surface while maintaining acceptable intensity of diffracted radiation at the detector. For example, with kaolinite clay, the 0.02

* The specific equipment described is Norelco.

inch vertical slit irradiates a specimen width of about 1.4 mm. A stepwise traverse in 1.4 mm. increments along the YY' axis provides a measure of variability on the specimen surface, while rapid oscillation over the full excursion length along the YY' axis provides an integrated reflected intensity for a specimen width of about 9 mm. The length of specimen irradiated varies with θ . Again, for the 002 kaolinite peak, which is convenient for fabric study, the length of specimen irradiated is about 12 mm.

III. PROCEDURES

One of the main problems in applying X-ray diffraction to fabric study is to obtain a flat surface for examination that contains the clay crystallites in the same orientation which exists inside a clay mass. Moreover, specimens for fabric study must be carefully marked so that the orientation of the specimen relative to the original engineering sample will be known. The optimum specimen size is about 20 mm. square and 6 to 8 mm. thick where either or both of the largest faces may be examined by X-ray diffraction. Larger specimens require a long time for impregnation and smaller specimens may seriously reduce the diffracted X-ray intensity.

In order to prepare a very flat surface for X-ray examination, the moist clay specimens are impregnated with Carbowax No. 6000. Carbowax is completely miscible with water when liquid at 65°C, and is a solid with hardness on Moh's scale of one at room temperature. Leaving the top surface of the specimen exposed to vapor the first day of immersion in liquid, carbowax allows entrapped gases to escape and prevents specimen rupture. Replacing the liquid carbowax with fresh wax every two or three days hastens the diffusion exchange of wax for water which results in firmer specimens. The exact time required to give sufficient exchange, wax for water, so that the specimen can be ground, will naturally vary depending upon the specimen: 1) size, 2) water content, and 3) bulk density. Ten days total immersion time, where the liquid carbowax was replaced two or three times, has generally proved satisfactory. At the completion of impregnation, the specimen should be removed from the liquid carbowax and allowed to slowly cool to room temperature. As mentioned, it is very important for X-ray diffraction that the surface to be examined be perfectly flat. Further, it is highly desirable that the opposite face be exactly

parallel to the flat surface to be X-rayed, because this greatly facilitates correct alignment in the pole figure device. To accomplish these requirements, a special jig was made from a triangular metal block 86 mm. on a side and 13 mm. thick. By proper setting of the three adjustable feet, one near each corner of the block, it is possible to grind the surface of a specimen flat and parallel to its opposite face. It is also possible to grind off either a known thickness and thus examine successive slices, or a known tip to the original reference plane. The specimen to be ground is held to the flat machined surface of the block by vacuum. Five 1 mm. holes over the specimen area of 400 mm² lead to a flexible vacuum line attached at the side of the block. A suitable trap system prevents grinding compound and kerosene from entering the vacuum pump.

The actual grinding is done with No. 600 carborundum powder in kerosene on a plate glass surface. In practice, one surface of a rough specimen is ground approximately flat while holding it in ones hand. This approximately flat surface is then placed in the jig and the opposite face ground flat. Usually two grindings of opposite faces result in excellent flat parallel faces for X-ray examination.

A Norelco X-ray diffraction unit equipped with a Geiger detector and a pole figure device was used. Unfiltered copper radiation was generated at 50 kv and 20 ma. The most satisfactory speeds for 2θ and chart during diffraction were found to be $1/4^\circ - 2\theta/\text{min.}$ and $1/4 \text{ in.}/\text{min.}$ respectively. Slits employed were: 1) 1° divergence and scatter, 2) 0.006 in. receiving, and 3) 0.02 in. vertical. For both α and ϕ rotations, the $20^\circ/\text{min.}$ drive motor was used which gave an α rotation of $20^\circ/\text{min.}$ and a ϕ rotation of $3-1/3^\circ/\text{min.}$ The peak ratios were slightly lower for the slower scanning speed, but the standard deviation was reduced from 30% to less than 10%.

The following procedures have been developed for obtaining ideal random and ideal oriented samples of kaolinite.

A. Recommended Procedure for Ideal Random

The ideal random condition is obtained from air-dried powdered clay carefully sieved to give a flat surface. After some experimentation, the following procedure was adopted. The sample holder is a brass cylindrical tube 15 mm. in diameter and 3 mm. high. The bottom of the cylinder is closed and has a flat surface which is exactly parallel to the top edge. Before filling the holder with the sample, the holder is first aligned in the pole figure device using the top edge of the holder as the reference plane. Air-dried powdered clay is sieved through a No. 200 sieve to produce a homogeneous sample. This uniform clay is then sieved through a No. 100 sieve over the sample holder until the holder is level full. A little practice and careful sieving gives a reasonably level surface and very good random particle arrangement. When the filled holder is again placed in the pole figure device, the specimen is ready for X-ray examination. In addition to determining the peak ratio, P.R., for the chosen 00 ℓ and hko reflections, both α and ϕ rotations should be made on at least one diffraction peak to ensure that the specimen is random.

B. Recommended Procedure for Ideal Orientation

1. Add 15 g. of kaolinite to 85 ml of distilled water (15% slurry).
2. Mix for 30 min. with a mechanical stirrer.
3. Adjust slurry to pH 11 ± 0.5 by addition of dilute NaOH solution.
4. Mix for 30 min. with a mechanical stirrer.
5. Place 0.770 ± 0.005 g. of slurry onto a 22 mm. square cover glass. The amount of slurry placed on the cover glass is controlled by the use of a medicine dropper that has been marked to give the proper volume.
6. Dry the slurry in a 50% relative humidity desiccator. The cover glasses must be level during the drying period. For kaolinite clay, the drying time varies between 20 and 30 hours.

The above procedure gives reproducible results with good orientation.

IV. RESULTS WITH KAOLINITE

The experiments on kaolinite clay with different fabrics were designed specifically to investigate the qualitative and quantitative aspects of X-ray diffraction as a method for evaluating clay fabric.

A. Choice of Peaks for Diffraction

The top diffraction trace in Fig. 2 illustrates the three main reasons for choosing the 020, ($2\theta = 20^\circ$) and the 002, ($2\theta = 25^\circ$) reflections to express the peak ratio, P.R. The 020 and 002 are: 1) strong reflections and thus provide a sensitive measure of fabric, 2) distinct from other reflections, and 3) close enough together that approximately the same area will be irradiated in the determination of the peak amplitudes for both peaks. Diffraction traces at the bottom of Fig. 2 indicate the method of measuring the peak amplitude. In actual practice, diffraction is recorded from $2\theta = 19.3^\circ$ to 20.1° and from $2\theta = 23.3^\circ$ to 25.1° . Running the diffractometer at $1/4^\circ$ 2θ /min. usually provides a well defined minimum from which to measure the amplitude, and since only about 2.6° 2θ are recorded, a single peak ratio is easily determined in 11 minutes.

The 002 peak is generally stronger than the 020 peak and also is much more symmetrical (see Fig. 2); therefore, the 002 peak was used for all α and ϕ rotations. For each specimen examined, the position of the 002 peak was determined and this value used for the α and ϕ rotation on that specimen. Depending upon the fabric, the 2θ value for the 002 peak may vary as much as 0.1° 2θ .

B. Statistics for Diffraction Peaks

Since one of the purposes in measuring fabric is to ascertain the variation within a specimen as well as between specimens, one needs to know how much variation arises from random fluctuations and sample preparation. To measure the effect of random fluctuations upon the peak ratio, the same identical area was examined ten times. Examination of peaks with different amplitude established

the random variation to be expected for peaks of different amplitude. For any surface where the standard deviation, S.D., of the observations exceeds that for random variation, the surface is heterogeneous. Table 1 gives the amplitudes and standard deviations as both the absolute value and as a percentage. Standard deviation, S.D., means that if one measured an amplitude of 40 counts/sec. (for which S.D. = 2 from Table 1), the chances are that 68 times out of 100 the amplitude of this peak would fall between 38 and 42. The per cent standard deviation in a dividend, such as the peak ratio, equals the square root of the sum of the squares of the per cent S.D. for the individual measurements. Therefore, if the peak ratio is 38(304/8), the per cent S.D. for this peak ratio is $\sqrt{101.7}$, or if one neglects the error in the 002 peak the per cent error in the peak ratio is $\sqrt{100}$. It is readily apparent that the larger the P.R. the more important becomes the error in the 020 peak. Statistical error over the surface of a specimen and between surfaces will be considered under each sample.

C. Effect of Impregnation

For the statistical probability to be useful as a measure of surface heterogeneity there must be no adverse effect upon the fabric from the carbowax impregnation. Mitchell (1956, Sc.D. Thesis, Department of Civil Engineering, M.I.T.) employing light microscopy found that impregnation with carbowax did not effect the fabric of a clay. To check further that impregnation with wax did not alter the fabric appreciably the following test was performed. A consolidated kaolinite sample at a water content of 75%* was sliced with a wire to give three flat surfaces. The surfaces were X-rayed in the wet condition and again after impregnation with

* The liquid limit for this clay is 105%.

carbowax. The results (Table 2) indicate less than 5% variation in peak ratio for any one of the three surfaces which is well within expected random variation. Since impregnation did not alter the fabric for the very severe condition of an exposed surface, it is extremely unlikely that impregnation would alter the fabric inside a clay mass.

D. Ideal Random

Initial tests were performed on specimens prepared according to the method of McCreery (J. Amer. Ceramics Soc., Vol. 32, p. 141, 1949) and examined in the standard diffractometer. Presieving the air-dry kaolinite powder lowered the peak ratio from 2.2 to 1.9. For the -1μ fraction of kaolinite, no change in P.R. occurred whether the flocculated slurry was dried from water or from acetone prior to sieving. In the pole figure device, specimens prepared by the McCreery method gave a very uniform spin, α rotation; however tip, ϕ rotation fell off rather quickly even with an 0.02 inch vertical slit.

Modifying the McCreery procedure by eliminating all packing gave a uniform ϕ rotation from $+15^\circ$ to -15° . In an effort to keep the loose powder from falling out during ϕ rotation, the surface was covered with a thin plastic film;* however, the plastic film raised the peak ratio back to 2.2 which was considered unsatisfactory.

Careful sieving to fill the sample holder gave a fairly smooth appearing surface and a peak ratio lower than had been obtained heretofore. Spin, α rotation (Fig. 3) shows that the specimen surface is reasonably flat and random since the variation

* The film was prepared by allowing a solution of 1 part collodion to 15 parts amylacetate to spread on water. The film thus formed was picked up on a 1 inch "O" ring and carefully placed over the clay specimen.

between samples A and B does not exceed the random variation obtained for three successive cycles upon the same surface.

The problem of the loose powder falling out upon ϕ rotation was not as serious as first supposed. No visible sign of surface disturbance was seen until a ϕ of 63 to 70° was reached; then the whole specimen just fell out. Figure 4 indicates that the variation in ϕ rotation between different specimens prepared at different times is no greater than the ϕ variation for different α positions on the same specimen. The gradual fall-off of intensity beyond about 15° arises from the failure of the pole figure device to maintain perfect diffraction geometry under these very extreme conditions. The rate of intensity decrease is twice as great for the larger vertical slit (0.04 in.).

The variability in the peak ratio between different specimens and within a particular specimen is quite small (Table 3). In fact, the variation in the 020 peak does not exceed expected random variation. Variability in the 002 peak is about 1.7 times (see Table 1) that expected from random variation which actually is considered quite good in view of the fact that the specimen surface was prepared by sieving alone with no further leveling.

E. Ideal Orientation

The recommended procedure for ideal orientation involves careful control of a number of variables. Further, changes in two or more of these variables may give as good or better orientation than obtained by the recommended procedure; however, the recommended procedure gives a very high peak ratio and consistent results.

Table 4 indicates the effect of slurry concentration upon the 002 peak amplitude. It is felt that where the 020 peak is so extremely weak, the variation in the 002 peak gives a more realistic picture of changes than would the peak ratio. Mixing by hand

consistently gave smaller 002 peaks than mixing with a mechanical stirrer. The time of mixing may be important because it was observed that sometimes stirring for 30 minutes gave decidedly low 002 peak amplitude. Hence, the 60 minutes is recommended; however, longer mixing times were not investigated.

The recommended amount of slurry (0.770 g.) on a cover glass is about all that will stay on. One-half to two-thirds of this amount of slurry definitely lowers the 002 peak amplitude. For the recommended procedure, the amount of clay on the cover glass is 24 mg./cm^2 which means that the clay film is about 90μ (0.035 in.) thick.

Drying conditions also influence the size of the 002 peak amplitude (Table 5). The difference between 50% R.H. and 71% R.H. is not significant; however, 50% R.H. consistently gave clay films where the S.D. of the 002 peak was lower than for 71% R.H.

Table 6 gives results for three batches of slurry prepared at different times by the recommended procedure. The 002 peak amplitude is significantly lower than for some of the conditions in Table 5; however, the clay films for the data in Table 5 were all prepared at the same time and the exact clay concentration, mixing condition, and amount of slurry on a slide are unknown. Haphazard attempts to repeat the optimum condition in Table 5 gave 002 peak amplitudes between 300 and 500 counts/sec. While there is more variation between batches than between samples in a batch, the data in Table 6 are considered satisfactory proof that the recommended procedure gives reproducible results. For Batch 1, Slide 2 and Batch 3, Slide 2, the S.D. over the whole slide does not exceed the expected random variation. While the percentage variation in the 020 peak between slides is large, in no instance does the variation exceed the expected random variation. Similarly, α rotation on the 002 peak of a good ideal orientation specimen will not fluctuate more than expected for random variation.

ϕ rotation on the 002 peak is virtually the same whether the amplitude is 650 or 750 counts/sec. However, as shown in Fig. 5, a sample dried at 100°C gives a much flatter curve as well as a lower peak height, indicating much poorer orientation. That the spreading of the curve for the oven-dried sample is more than just a lower peak height is shown in Fig. 6. The ϕ for the bottom surface has the same shape as the ϕ curve for the top surface, the only difference being that the bottom surface has a peak height of 1/4 that of the top surface. Figure 6 compares ϕ curves for ideal random and ideal orientation samples. The difference is obvious. It is interesting that at large ϕ the slope is the same for both which is to be expected from the diffraction geometry. For ideal orientation, the ϕ curve is constant irrespective of α position. Figure 5, Sample A, shows the maximum variation found in ϕ curves for six different α positions.

A peak ratio of 21 for the bottom surface of an ideal orientation sample, where the top surface has a P.R. of 270, probably represents segregation of the clay during drying because the ϕ curve for the bottom surface indicates that the orientation is excellent. Therefore, the coarse clay gives an ideal orientation P.R. of 21 and the fine clay an ideal orientation P.R. of 270.

Ideal oriented clay films mounted with 00 ℓ normal to the X-ray beam gave a P.R. by transmission rather than reflection of 0.1 ± 0.3 . The large error is a combination of the small 002 peak and the difficulty of mounting the clay films.

F. Isotropic Consolidation

A flocculated kaolinite slurry at a water content of 190% was placed in a balloon, evacuated very slowly for 2 months, placed in a triaxial cell, and consolidated isotropically to 1 kg./cm². The final water content was 50%. After consolidation, the clay sphere

was cut into quarters and the pieces placed in carbowax. Sections for X-ray examination were prepared approximately parallel and normal to the direction of general seepage which was assumed to be out through the opening in the balloon.

Table 7 summarizes the P.R. for isotropic consolidation. The effect of surface disturbance is clearly evident in the much higher peak ratios for the original surface (thickness X) which results from the original cutting of the wet clay before it was immersed in wax. The average P.R. for subsequent slices of the carbowaxed clay parallel to seepage is 1.65 ± 0.10 which is only a 6% variation and does not differ significantly from ideal random. The P.R. for the specimen normal to seepage is 2.6 which is significantly higher than for the specimen parallel to seepage.

While the average P.R. between slices shows a very modest variation, there was considerable variation over a given surface. The peak ratio range in Table 8a is from 1.14 to 2.16. The observed standard deviation, S.D., on the X-0.06 inch surface for the 002 and 020 peaks are four times and two times respectively that expected from random variation for peaks of the appropriate amplitude. Making use of the standard deviation for random variations, S.D.R., for peaks of the same amplitude, considerable detail upon a particular surface can be detected. In Table 8a at $\alpha = 0$, the P.R. is very definitely high (more orientation at this spot) because the 002 peak is 2.5 times S.D.R. above the average for this surface and at the same spot the 020 peak is 3 times S.D.R. below the average for this surface. The same surface at $\alpha = 175$ gives a very low P.R. compared to the average for this surface. The decrease in the 002 peak should be accompanied by an increase in the 020 peak if an orientation change was the only factor contributing to the change in the 002 and 020 peaks. The 002 peak is

5 times S.D.R. below the average for this surface while the 020 has not increased at all which strongly suggests some other factor is involved. A decrease in clay concentration at the $\alpha = 175$ position would lower the amplitude of both 002 and 020. Therefore, at the $\alpha = 175$ position there must be a decrease in clay concentration as well as a lower peak ratio. For $\alpha = 250$, in Table 8b, there is definitely a lower clay concentration because both the 002 and 020 peaks are way below the average (5 times S.D.R. for 002 and 4 times S.D.R. for 020) while the P.R. remains unchanged. At $\alpha = 180$, in Table 8b, the peak ratio is definitely low because the statistical significant decrease in the 002 peak is accompanied by a statistical significant increase in the 020 peak.

It may be noted that for X-0.07 at $\alpha = 180$, the P.R. of 1.45 is conclusively low while for X-0.06 at $\alpha = 175$, which also had a much lower P.R., the certainty in this low P.R. is less because of the apparent change in clay concentration.

Both the α and ϕ curves provide additional evidence for significant differences in P.R. and/or clay concentration on a particular surface. The depression at $\alpha = 180$ for both α curves in Fig. 7a confirms the lower orientation already discussed for this α position. The low clay concentration at $\alpha = 250$ for X-0.07 likewise is confirmed in Fig. 7a by the pronounced minimum at $\alpha = 250$. This same minimum again shows up in the ϕ curves in Fig. 8. The marked depression in the α curve for thickness X in Fig. 7b must represent a very low clay concentration because the P.R. is the highest, yet the 002 peak has only two-thirds the amplitude it does at $\alpha = 40$.

The ϕ curves in Fig. 9 provide additional evidence that flocculated kaolinite slurry isotropically consolidated to 1 kg./cm² has a nearly random fabric because of the small difference in the

curves for isotropically consolidated clay and ideal random powder. The extremely close agreement between ϕ curves for parallel and normal to seepage, while the peak ratio changed by a factor of 2, suggests that the P.R. is the more sensitive measure of fabric.

G. Anisotropic Consolidation

The same flocculated kaolinite slurry used for isotropic consolidation was used for anisotropic consolidation. A flocculated slurry at a water content of 190% was consolidated one-dimensionally to 8 kg./cm^2 in a special oedometer of 3 in. diameter. The specimen was then trimmed to fit a 7/8 in. diameter oedometer and further consolidated in small increments up to a total stress of 197 kg./cm^2 . Because of the rather large side friction in such a small oedometer cell, the actual consolidation pressure is not known. At the completion of consolidation the clay was immersed in carbowax, and finally, specimens cut at approximately 0, 45, and 90° to the direction of the consolidation pressure for X-ray examination.

The average peak ratios, P.R., for specimens at 0, 45, and 90° to the direction of the consolidation pressure, $\bar{\sigma}_c$, are 0.8, 2.8, and 26 respectively. Whether or not flocculated kaolinite consolidated anisotropically to high pressure should be considered well along toward ideal orientation depends upon the reference used. If one uses a P.R. of 270, as obtained for the top surface of an ideal orientation, then a P.R. of 26 is not far along; however, if one uses a P.R. of 21 as obtained for the bottom surface of an ideal orientation, then a P.R. of 26 is extremely well oriented. Figure 12 confirms that this high $\bar{\sigma}_c$ has produced a very well oriented clay fabric.

The homogeneity of the specimen surface may be investigated for both fabric variation (P.R.) and clay concentration (density changes). Experimental measurements required are amplitudes of 002 and 020 peaks for different α and osc. positions as well as α

and ϕ rotations on the 002 peak. In Table 9a the observed standard deviation, S.D., in the 020 peak of 1.2 is close to the expected random variation, S.D.R., (refer to Table 1) of 1.0 for a peak of this amplitude. At the same time, the observed S.D. in the 002 peak is 80 which is twenty times S.D.R. for a peak of this size. Obviously the surface is heterogeneous. Comparison of 002 and 020 peaks at $\alpha = 40$ and 330 , indicates that the improved orientation (higher P.R.) at $\alpha = 40$ is accompanied by an increase in clay concentration. Such an increase in clay concentration might be expected because the better the orientation (the higher the peak ratio) the smaller will be the void volume, and consequently the higher will be the clay concentration.

Table 10 gives amplitudes of 002 and 020 peaks for various positions and a traverse across the specimen surface cut at 0° to $\bar{\sigma}_c$. The ab plane of the clay crystallites lies approximately parallel to the direction of the primary X-ray beam when $\alpha = 90$ (see sketch in Fig. 13). The observed S.D. for 002 is 1.0 and S.D.R. = 1.0 for 002, while for the 020 peak the observed S.D. = 4.1 and S.D.R. is 1.5; therefore, since the observed variation in the 020 peak is three times S.D.R., one can be 99% sure that the surface is heterogeneous. For $\alpha = 60$, the 020 peak is clearly stronger than the average for this surface since the probability is 99% that the amplitude of 39 exceeds the expected average value. If there were no change in clay concentration this increase in 020 should be accompanied by a decrease in the 002 peak because the sample is at 0° to $\bar{\sigma}_c$; however, the 002 peak has not decreased. Therefore, there is an increase in clay concentration as well as a lower P.R. at this spot. At $\alpha = 180$ the opposite effect is observed and is consistent with what should be expected for a sample cut at 0° to $\bar{\sigma}_c$. It should be noted that an increase in clay concentration for a specimen surface at 0° to $\bar{\sigma}_c$ gives a decrease in P.R., the opposite is true for a specimen surface at 90° to $\bar{\sigma}_c$.

Comparison of α rotation on the 002 peak for 0° to $\bar{\sigma}_c$ and the measured amplitudes of 002 peaks for the same identical surface mounted in exactly the same position at first glance seem to contradict one another. The maximum change in 002 amplitude is only 2 counts/sec. between $\alpha = 0$ and $\alpha = 180$, while the α rotation shows a change of 10 counts/sec. in the 002 peak between $\alpha = 0$ and $\alpha = 180$. This apparent discrepancy is in reality just another proof that there is a change in orientation at $\alpha = 180$ and the resultant tipping of the clay crystallites shifts the 2θ maximum of the 002 peak. The α rotation is for 2θ fixed on the 002 peak as measured at $\alpha = 0$, and when $\alpha = 180$ the diffraction maxima may not be at the same 2θ as for $\alpha = 0$. Reference to Fig. 2 shows that a change in 2θ of only 0.05° would change the peak height by 15%.

On the traverse (Table 10a) the observed S.D. for both 002 and 020 peaks is more than two times the expected random variation, S.D.R.; therefore, these data indicate a better than 95% probability that the surface is heterogeneous in fabric and/or clay concentration. Positions R + 2 and R + 7.5 have 002 and 020 peaks more than two times S.D.R. below the average for the surface; however, the P.R. for both does not deviate significantly from the average. Accordingly, these lower peaks are ascribed to a decrease in clay concentration. For this situation where the P.R. has not changed, one can estimate the change in clay concentration from the change in amplitude of the 002 and 020 peaks. Assuming that clay concentration is proportional to amplitude, the 002 peak is low by 12% at R + 2 and the 020 peak is low by 11% at the same direction. At R + 7.5, the 002 peak is low by 8.5% and the 020 peak by 9.5%. The R + 6.5 position has a higher clay concentration indicated by: 1) no change in P.R., 2) an increase in 002 by 12%, and 3) an increase in 020 by 10%. Here again the change is two times S.D.R. or 95 chances out of 100 that this spot has a high clay concentration. At R + 4.3 the 002 peak is high and the 020 peak low giving a 90 out

of 100 chances that this spot has a higher P.R., i.e., is better oriented.

The sample cut at about 45° to $\bar{\sigma}_c$ also appears to be heterogeneous as evidenced by the data in Table 9b and Fig. 10.

ϕ rotation on the 002 peak for anisotropically consolidated kaolinite cut at 90° to $\bar{\sigma}_c$ (Fig. 11) indicates that the specimen surface is not exactly at 90° to the orientation of the clay crystallites because: 1) at $\alpha = 0, 260$, and 300 the peak in ϕ rotation occurs at -4° not at 0° , and 2) at $\alpha = 180$ there is a significant increase in height of the ϕ curve and the peak is now at 0° . Grinding off 0.025 inches did not change the ϕ curve. Figure 12 gives a comparison between ideal orientation and one-dimensional consolidation at 90° to $\bar{\sigma}_c$. Clearly, the amount of orientation produced by anisotropic consolidation of flocculated kaolinite can be very marked.

ϕ rotation on anisotropically consolidated samples cut at 0 and 45° to $\bar{\sigma}_c$ are shown in Figs. 13 through 15. The sketches in Fig. 13 and Fig. 14 are included as an aid to understanding the ϕ curves. Shading indicates the approximate orientation of clay crystallites and the small dotted square at the left represents the 00 l face of a single clay crystal (drawn on an exaggerated scale). The angle between clay crystallites and the vertical for the 0° to $\bar{\sigma}_c$ condition was deliberately exaggerated to clearly indicate which way the crystallites were tipped. Arrows on the surface reveal the direction of the X-ray beam for different α positions. The angle β shows from which side of the specimen surface a tip was deliberately ground.

For samples cut at 0 and 45° to $\bar{\sigma}_c$, the ϕ curves change drastically depending upon the α position. The interrelationship between the ϕ curves and the sketches in Figs. 13 and 14 is just what would be expected. At $\alpha = 0$ and 180 , the ϕ curves are identical and very nearly symmetrical. The extreme ϕ curves occur at $\alpha = 90$ and 270 which are very asymmetric and obviously different because

of the different orientation of the $00l$ face of the clay crystallites at $\alpha = 90$ or 270 . If for the ϕ curve at $\alpha = 270$, positive ϕ is plotted negative and negative ϕ is plotted positive the resultant curves gives an excellent fit with the $\alpha = 90$ curve. This mirror image relation between the ϕ curves at $\alpha = 90$ and 270 is related, of course, to the angle that the $00l$ crystal plane makes with the normal to the surface being examined.

Ideally, if the angle between the $00l$ crystal plane and the normal to the surface were 0° then the ϕ curve would have a minimum at $\phi = 0$; however, because of the difficulty in maintaining perfect diffraction geometry during ϕ rotation the diffraction intensity falls off (see Fig. 4). In Fig. 13 at $\alpha = 90$ or 270 the curve tends toward a horizontal line, while at $\alpha = 180$ the slope at large ϕ is flat (7.6 counts/sec. per $20^\circ \phi$) compared to ideal random (11 counts/sec. per $20^\circ \phi$).

Figure 15 shows the effect upon the ϕ curve for 0° to $\bar{\sigma}_c$ when the angle between the $00l$ crystal plane and the normal to the irradiated surface is increased a few degrees. The only significant change in the ϕ curves for the 4° change in the irradiated surface* is the peak at $\phi = 0^\circ$ when $\beta = 2^\circ$. The average P.R. for $\beta = 0, 3$, and 4° is 0.72 and all three values are well within the S.D. of 0.1 established in Table 10b. The S.D. indicates a 99% probability that the P.R. of 1.0 for $\beta = 2^\circ$ is a real difference and did not arise by chance. Again, the P.R. and ϕ curve are in complete accord.

The 2.5° angle ground on the surface of the specimen cut at 45° to $\bar{\sigma}_c$ decreased the angle between the $00l$ plane and the normal to the irradiated surface which should decrease the P.R. At $\alpha = 90$ the P.R. went from 3.9 when $\beta = 0$ to 2.7 when $\beta = 2.5^\circ$ which

* The specimen is mounted so that its surface still lies exactly in the XY plane of the pole figure device.

suggests that at this angle between the 00 ℓ crystal plane and the surface, a slight change in the angle produces a decided change in P.R. However, the ϕ curves for $\alpha = 90$ (Fig. 14) reveal that while the change in P.R. at $\phi = 0$ is definitely real, the cause of the change in P.R. at $\phi = 0$ arises from a secondary orientation feature and not from a general change in orientation. Clearly, the general shape of the ϕ curves at $\alpha = 90$ for both $\beta = 0^\circ$ and $\beta = 2.5^\circ$ are very much the same. The P.R. at $\phi = -43^\circ$ is 19 when $\beta = 0$ and is 22 when $\beta = 2.5^\circ$. Both of these results are within the peak ratio range determined on the specimen cut at 90° to $\bar{\sigma}_c$.

H. Unconfined Shear

The sample for unconfined shear (3/4 in. diameter by 1.5 in. high) was cut from a large block of flocculated kaolinite that had been consolidated one-dimensionally to 2 kg./cm². The water content of the kaolinite at the time of shear was 44%. After shear, the whole sample was immersed in carbowax for a total of 3 weeks. The carbowax was replaced every week. Figure 16 shows the relation of the X-ray specimens to the shear sample and the position of the spots actually X-rayed. Specimens A and B were marked carefully so that after each grinding the sample was mounted in exactly the same position.

Both the top and bottom surfaces of Section B, which was at 90° to both $\bar{\sigma}_c$ and the deviator stress during shear, were X-rayed. Since the standard deviation for random variation at a P.R. of 9 is ± 1 , the data in Table 11 show that there was no significant change in P.R. for successive slices and, further, the difference between the top and bottom surfaces has a low level of significance. The α curves (not shown) on both top and bottom surfaces are very uniform confirming the uniform P.R. Likewise, the ϕ curves (not shown) were all virtually identical symmetrical curves for: 1) the top and bottom surfaces, 2) successive slices, and 3) α position of 0° and of 90° .

A series of 25 successive slices through the shear zone (Section A) were examined. The results are summarized in Fig. 17. The peak ratios for the four spots were averaged and if at least three of the ratios were within the 95% confidence limit (2 S.D.R.), the average P.R. was plotted. When one P.R. on a given slice exceeded the 95% confidence limit it was plotted separately. If more than one P.R. exceeded the 95% confidence limit then all four P.R. were plotted separately for that particular slice. It is readily apparent from Fig. 17 that: 1) there is more than one region of marked particle orientation in the shear zone, 2) the shear zone is not planar, 3) the maximum orientation in the shear zone (about 30° to $\bar{\sigma}_c$) is at least as large as for the surface at 90° to $\bar{\sigma}_c$ (Section B) but outside the shear zone, and 4) the variation in P.R. from spot to spot on the surface is greatest when particle orientation is greatest. Between thickness 0.11 and 0.14 in. in Fig. 17, the real maximum in particle orientation was probably missed because of the arbitrary removal of the 0.01 in. slices.

Figures 18 and 19 show the maximum variation observed in the ϕ curves for the 25 slices at spots 2 and 4 which are at 90° to each other. The ϕ curves for spot 1 are very similar to those for spot 2 and the ϕ curves for spot 3 closely resemble those of spot 4. As one might expect, the ϕ curves in Figs. 18 and 19 have the same general shape as those for anisotropically consolidated kaolinite cut at 45° to $\bar{\sigma}_c$ (Fig. 14). The ϕ curves of Fig. 19 reveal the improved orientation at thickness 0.121 in. and 0.083 in. because of the increased amplitude and equally important, the decreased width at half amplitude of the curves. While the amplitude of the ϕ curves in Fig. 18 does not change tremendously, there is a very marked shift in the position of the ϕ curve peak in the region of maximum shear. This shift in the peak of the ϕ curve gives rise to the marked increase in P.R. because the P.R. generally is

measured at $\phi = 0$. Again, in Fig. 18 the width of the ϕ curve at half amplitude is smaller the better the particle orientation. When the ϕ curve at spot 2 on a surface is very asymmetrical, indicating that the surface is outside the shear zone, the P.R. measured at $\phi = +30^\circ$ is between 9 and 10 and is in good agreement with the P.R. on Section B of the sheared clay sample.

V. DISCUSSION

The results of the fabric measurements on kaolinite amply demonstrate that a quantitative measure of fabric and sample heterogeneity can be achieved. The average P.R. for a particular surface is a quantitative expression of the particle orientation. The standard deviation for the same surface relative to the standard deviation for random variation quantitatively expresses heterogeneity of particle orientation on the surface. As mentioned for data on isotropic and anisotropic consolidated kaolinite, variations in clay concentration in the surface can be measured which is the other major factor in sample heterogeneity. One must always keep in mind that the area examined contains a very large number of clay particles.

The pole figure device, while helping locate the zone of maximum or minimum particle orientation and to obtain data for statistical variation, also provides valuable cross checks. These cross checks include any two or all three of P.R., α curve, and ϕ curve; thereby substantiating or refuting a tentative conclusion one may have reached based upon one of the measured variables.

Figure 20 is a tentative suggestion for a possible way to summarize a lot of fabric data so that one quickly views the overall quantitative picture. Clearly, isotropic consolidation to 1 kg./cm^2 is fairly random and anisotropic consolidation to 197 kg./cm^2 is well oriented. The unconfined sheared sample is moderately oriented and the shearing action clearly produces orientation at least as good as consolidation to an equivalent stress.

The log scale ordinate in Fig. 20 was arbitrarily chosen in order to more easily plot the data. Similarly, the linear nature of the plot is completely arbitrary. In fact, for ideal random, all the experimental data is for 90° to $\bar{\sigma}_c$ where the consolidating force is gravity. The lower end of the ideal orientation area is based upon experimental error in measurement of the P.R. while the upper end

of the ideal orientation area is determined by the peak ratios measured on the bottom and on the top surface of a specimen prepared according to the standard procedure for ideal orientation. It should be remembered that ideal random and ideal orientation are arbitrarily defined in terms of laboratory treatments and are intended as a relative scale against which engineering samples can be prepared. Much more data is needed before the shape of the plot P.R. versus angle to $\bar{\sigma}_c$ can be defined.

VI. RECOMMENDATIONS

Considerable refinement on several phases of the described method are possible; however at this stage it is believed that the procedure is adequate to warrant a detailed study of consolidation and shear. Specifically, kaolinite samples 1) consolidated to different pressures from various physiochemical environments, and 2) identical triaxial specimens sheared to different points along the stress-strain curve should be subjected to thorough fabric study. These data should not only prove valuable in themselves, but should greatly facilitate determining which phases of the procedure are worthwhile refining.

REFERENCES

1. Brindley, G. W., Min. Magazine, V. 30, p. 71, 1953.
2. Kubiena, W. W. et al, Zeit. Pflany. Ding., Boden, 1961.
3. Lambe, T. W., A.S.C.E. Proceedings No. 1654, 1958.
4. Mitchell, J. K., Highway Research Board Proceedings, V. 35, p. 693, 1956.
5. Olsen, H. W., 9th Clay Conference (In Press) 1960.
6. Raithburd, C. M., Conference on Research and Use of Clays, Published by Lvov University, p. 779, 1958.
7. Raithburd, C. M., Doklady K sobraniyu Mezhdunarodnoy komissii polizucheniya glin: Akad. Nauk. CCCP, Kom no Izucheniya Glin Otdel, Geol. -Geograf. Nauk., Moscow, pp. 108-117, 1960.
8. Seed, H. B. and Chan, C. K., Transactions A.S.C.E., V. 126, p. 1344, 1961.
9. Van Olphen, H., Thesis, Delft University, 1951.

TABLE 1

RANDOM VARIATION IN PEAK AMPLITUDE

Peak	Amplitude (counts/sec.)	S.D.* (counts/sec.)	S.D.* (%)
020	2.5	0.5	20
020	5	0.8	16
020	8	0.8	10
020	14	1.1	8
020	17	0.9	5
020	29	1.1	4
002	35	1.6	5
002	42	1.9	5
002	128	3.5	2.7
002	170	4	2.3
002	300	4	1.3
002	640	8	1.3

* S.D. = Standard deviation, for 10 repetitions on the same identical area of specimen surface.

$$S.D. = \sqrt{\frac{\sum (\bar{m} - m)^2}{n - 1}}$$

where m = each observed amplitude and \bar{m} = mean amplitude of n observations.

TABLE 2

EFFECT OF IMPREGNATION WITH CARBOWAX
UPON THE PEAK RATIO OF KAOLINITE

Surface	Peak Ratio	
	H ₂ O	Carbowax
A	6.0	6.5
B	6.2	5.7
C	8.8	9.9

TABLE 3

IDEAL RANDOM KAOLINITE

Variation Within a Specimen				Variation Between Specimens			
Amplitude (counts/sec.)				Amplitude (counts/sec.)			
α°	002	020	P.R.	Specimen No.	002	020	P.R.
0	35.0	18.0	1.94	1	35.5	20.0	1.78
60	35.0	20.5	1.71	2	37.3	21.3	1.75
90	39.0	20.0	1.95	3	32.0	18.5	1.73
120	34.0	20.5	1.66	4	<u>32.4</u>	<u>19.5</u>	<u>1.66</u>
180	31.0	20.0	1.55	Ave. 34.3 19.8 1.73			
210	40.0	20.0	2.00	S.D. of mean (1.73) = 0.06			
240	37.0	22.0	1.68				
300	<u>34.0</u>	19.0	1.79				
330	<u>34.0</u>	<u>20.0</u>	<u>1.70</u>				
Ave.	35.5	20.0	1.78				
S.D.	2.8	1.1	0.14				

$$\frac{35.5}{20.0} = 1.78 \pm 0.17$$

S.D. = Standard deviation.

TABLE 4

EFFECT OF SLURRY CONCENTRATION ON
IDEAL ORIENTATION OF KAOLINITE

Slurry Concentration (g. clay/100 g. slurry)	Amplitude of 002 Peak (counts/sec.)	Standard Deviation for 002
6.5	525	11
10	560	60
15	870	9
25	735	20

TABLE 5

EFFECT OF DRYING CONDITION ON IDEAL
ORIENTATION OF KAOLINITE

Drying	Amplitude 002 Peak (counts/sec.)	Standard Deviation of 002
100°C	295	10
65°C	370	20
25°C, Room air	745	38
25°C, 50% R.H.	870	9
25°C, 71% R.H.	865	10

TABLE 6

REPRODUCIBILITY OF RECOMMENDED PROCEDURE
FOR IDEAL ORIENTATION OF KAOLINITE

Sample	002 Peak (counts/sec.)		020 Peak (counts/sec.)	
	Amplitude	S.D.	Amplitude	Peak Ratio
Batch 1, Slide 1	683	13	2.67	256
Batch 1, Slide 2	678	6	2.17	312
Batch 2, Slide 1	640	22	2.50	267
Batch 2, Slide 2	622	14	2.25	276
Batch 3, Slide 1	648	19	2.30	281
Batch 3, Slide 2	<u>637</u>	<u>8</u>	<u>2.41</u>	<u>265</u>
Average	645	25	2.40	270

TABLE 7

PEAK RATIO FOR ISOTROPICALLY CONSOLIDATED KAOLINITE*

Section Parallel to Seepage		Section Normal to Seepage	
Thickness (inch)	P.R.	Thickness (inch)	P.R.
X	3.50	X	3.12
X - 0.02	1.63	X - 0.01	2.66
X - 0.03	1.73	X - 0.03	2.56
X - 0.05	1.53		
X - 0.06	1.63		
X - 0.07	1.71		

* $\bar{\sigma}_c = 1 \text{ kg./cm}^2$; $w = 50\%$.

TABLE 8

PEAK AMPLITUDES AND PEAK RATIOS FOR
ISOTROPICALLY CONSOLIDATED KAOLINITE

(Section Parallel to Seepage)

A. Thickness X - 0.06"

α	Amplitude (counts/sec.)		
	002	020	P.R.
0	45.5	21.0	2.16
60	43.0	25.0	1.72
120	34.5	25.0	1.38
175	29.5	26.0	1.14
250	43.5	27.0	1.61
320	<u>45.0</u>	<u>25.5</u>	<u>1.76</u>
Average	40.2	24.9	1.63
S.D.	7.4	2.3	0.38

B. Thickness X - 0.07"

α	Amplitude (counts/sec.)		
	002	020	P.R.
0	42.0	23.0	1.82
60	42.5	24.5	1.73
120	37.0	20.0	1.85
180	32.5	25.5	1.45
250	28.5	17.5	1.68
320	<u>38.0</u>	<u>22.0</u>	<u>1.73</u>
Average	36.8	22.1	1.71
S.D.	6.1	3.5	0.16

TABLE 9

PEAK AMPLITUDES AND PEAK RATIOS FOR
ANISOTROPICALLY CONSOLIDATED KAOLINITE

A. Sample Cut at 90° to $\bar{\sigma}_c$ (horizontal surface)

α	<u>Amplitude (counts/sec.)</u>		Peak Ratio
	002	020	
40	370	11.0	34
120	350	11.0	32
240	238	12.5	19
290	270	10.0	27
330	<u>210</u>	<u>10.0</u>	<u>21</u>
Average	288	11.0	26
S.D.	80	1.2	8

B. Sample Cut at 45° to $\bar{\sigma}_c$

α	<u>Amplitude (counts/sec.)</u>		Peak Ratio
	002	020	
0	56	15.0	3.73
60	46	18.5	2.49
120	50	17.5	2.86
190	43	14.0	3.07
220	48	20.0	2.40
260	<u>45</u>	<u>19.5</u>	<u>2.30</u>
Average	48	17.4	2.76
S.D.	.5	2.0	0.43

TABLE 10

PEAK AMPLITUDES AND PEAK RATIOS FOR ANISOTROPICALLY
CONSOLIDATED KAOLINITE (0° to $\bar{\sigma}_c$ Vertical Surface)

A. α Positions

α	<u>Amplitude (counts/sec.)</u>		Peak Ratio
	002	020	
0	26.0	33.0	0.79
60	25.5	39.0	0.65
120	26.0	33.5	0.78
180	24.0	28.0	0.86
240	24.5	33.5	0.73
300	<u>24.0</u>	<u>30.5</u>	<u>0.79</u>
Average	25.0	32.9	0.76
S.D.	1.0	4.1	0.10

B. Traverse at $\alpha = 90$

Oscillating Position (mm.)	<u>Amplitude (counts/sec.)</u>		Peak Ratio
	002	020	
R + 0	28.5	37.5	0.76
R + 2.0	25.0	31.5	0.79
R + 3.0	29.0	38.0	0.76
R + 4.3	31.0	32.5	0.95
R + 6.5	30.5	40.0	0.76
R + 7.5	26.0	32.0	0.81
R + 8.7	<u>28.5</u>	<u>36.0</u>	<u>0.79</u>
Average	28.4	35.4	0.80
S.D.	2.4	3.5	0.10

TABLE 11

PEAK RATIO FOR CONSOLIDATED SHEARED KAOLINITE
CUT AT 90° TO THE DEVIATOR STRESS*

Thickness (in.)	Peak Ratio	
	Top	Bottom
X	7.8	10
X - 0.01	7.5	9.2
X - 0.02	7.6	10.2
X - 0.03	<u>9.1</u>	<u>9.7</u>
Average	8.0 ± 0.5	9.7 ± 0.5

* Section B, see text for explanation.



FIGURE 1 POLE FIGURE DEVICE MOVEMENTS

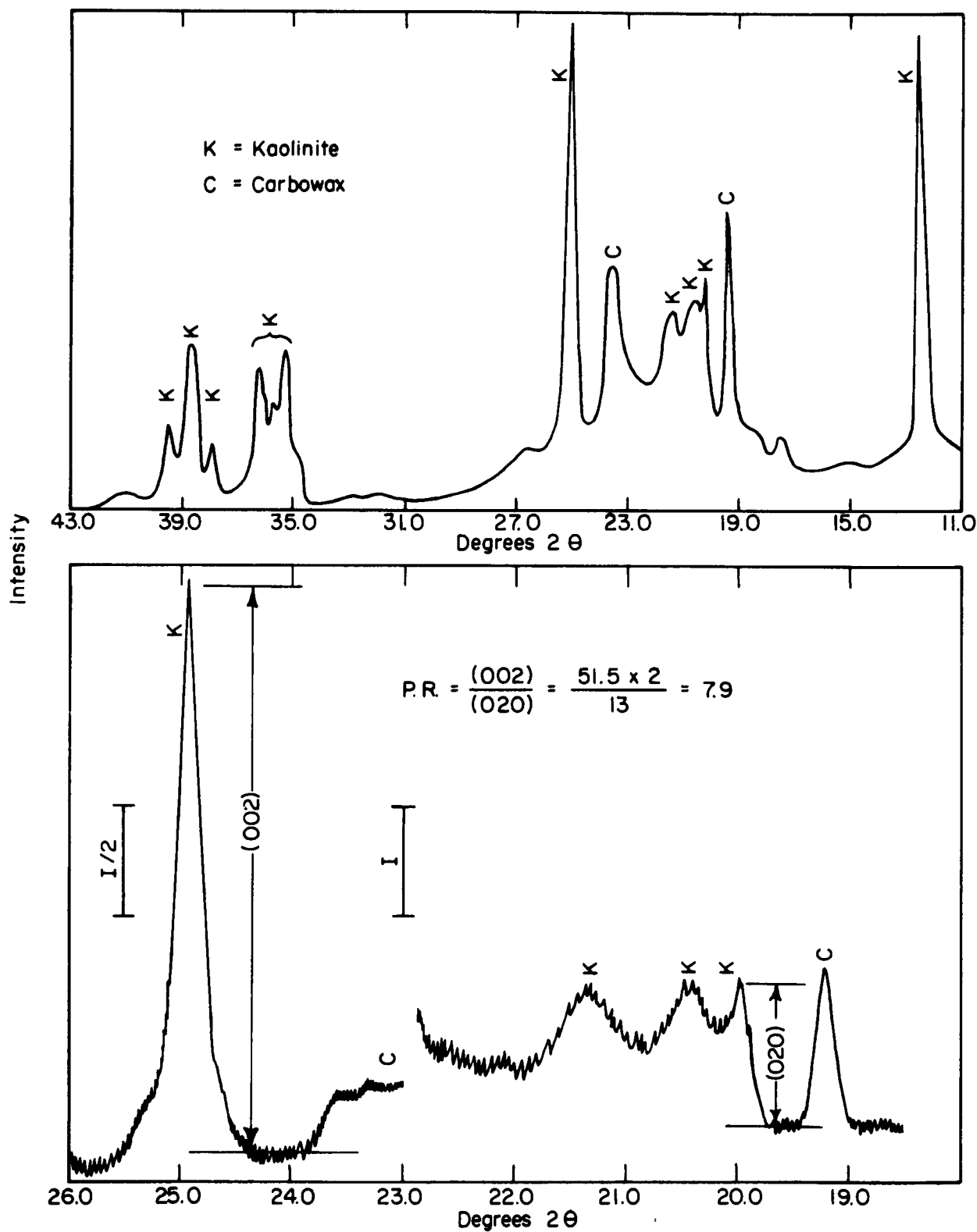
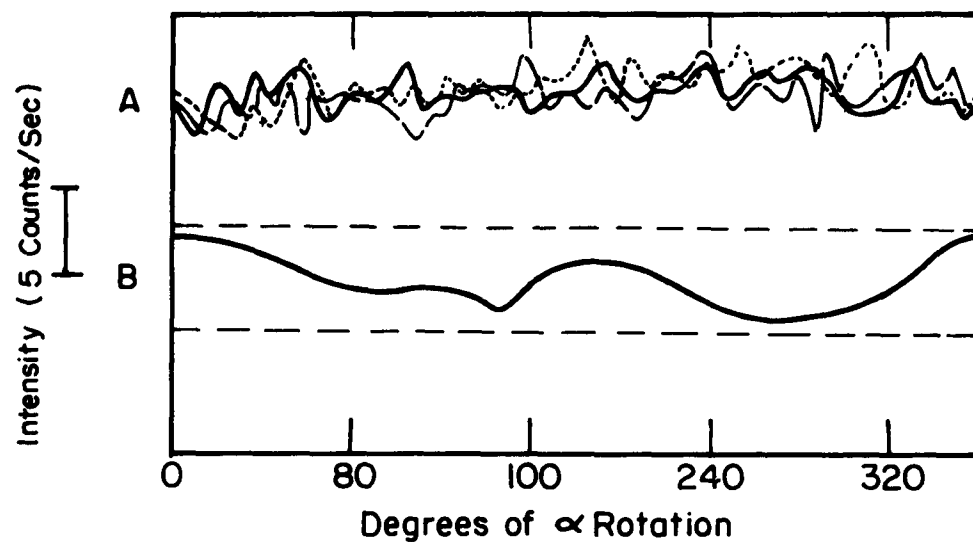


FIGURE 2

DIFFRACTION TRACES FOR KAOLINITE



A. Surface prepared by McCreery method

—— 1st cycle

----- 2nd cycle

--- 3rd cycle

B. Surface prepared by sieving

----- Indicates random variation
from A

FIGURE 3 α ROTATION ON 002 PEAK FOR IDEAL
RANDOM KAOLINITE

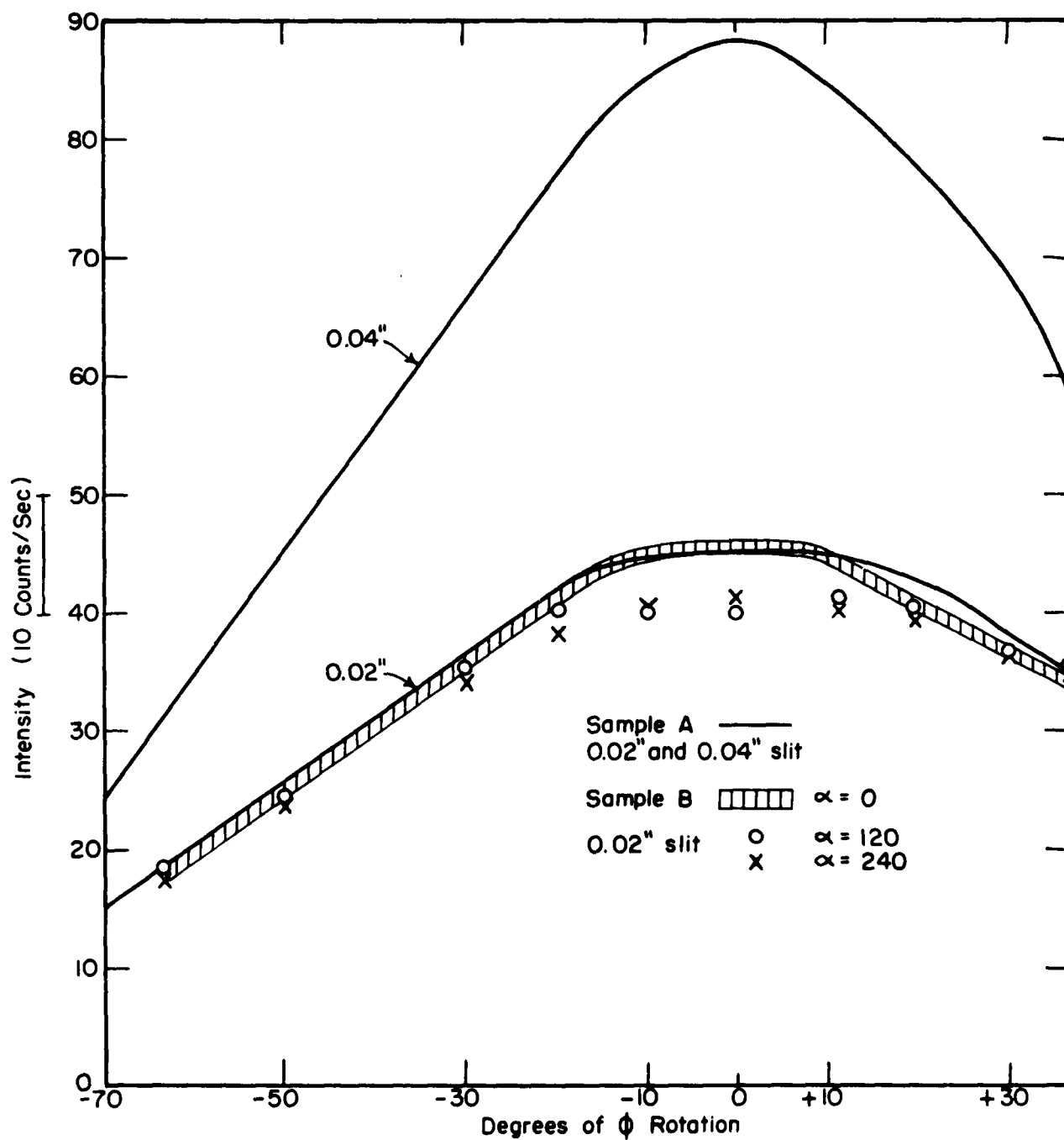


FIGURE 4 ϕ ROTATION ON 002 PEAK FOR IDEAL RANDOM KAOLINITE

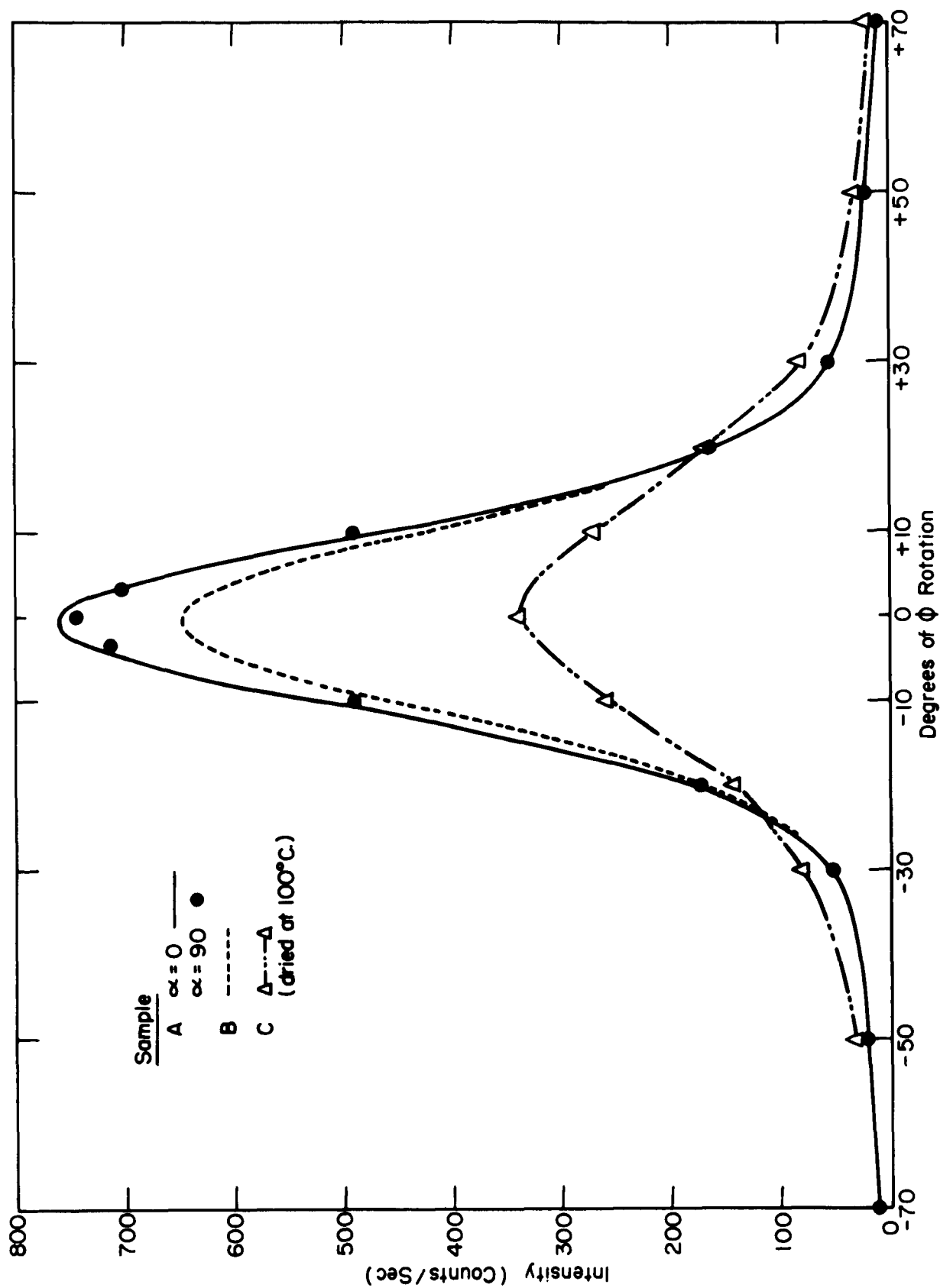


FIGURE 5 ϕ ROTATION ON 002 PEAK FOR IDEAL ORIENTED KAOLINITE

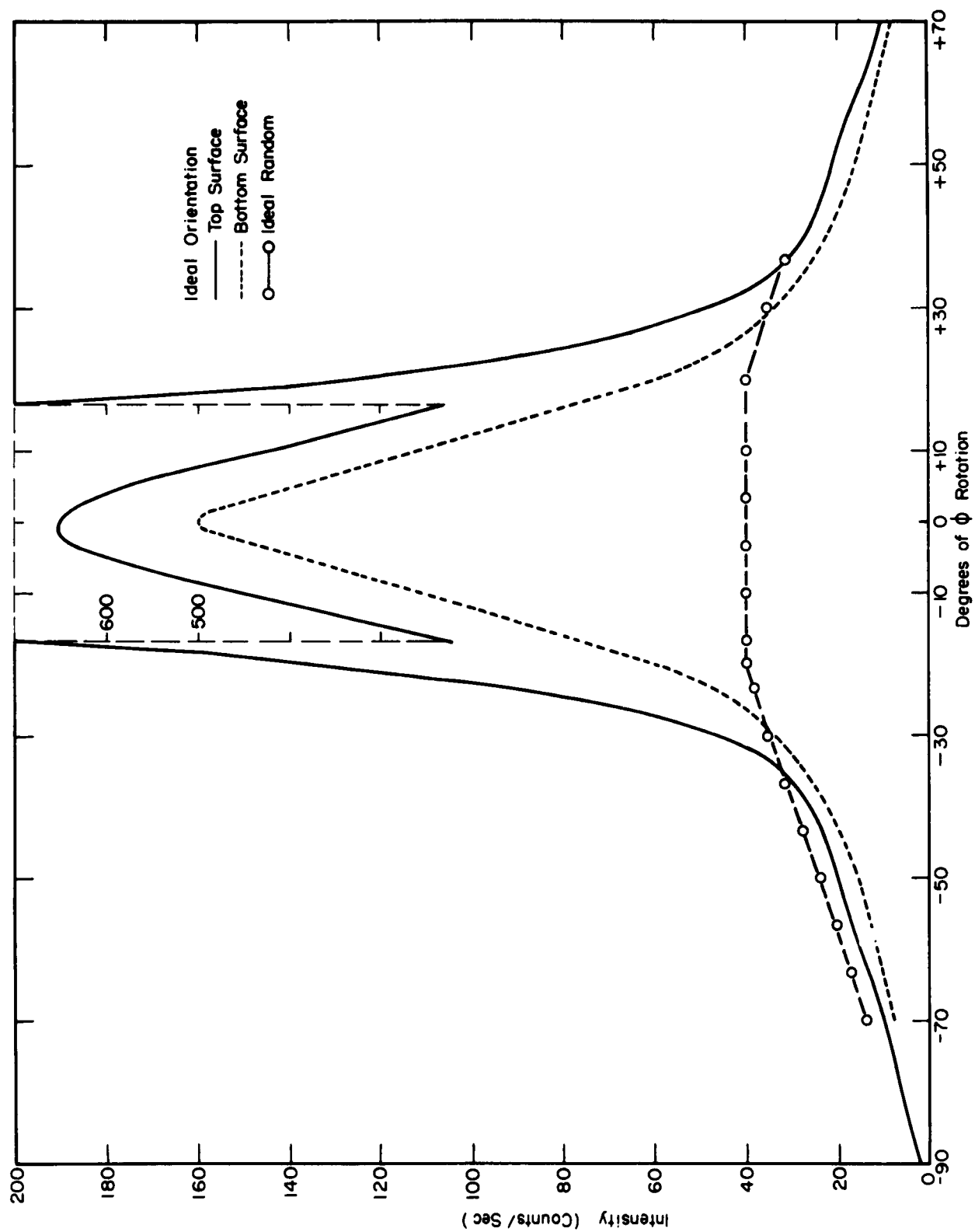
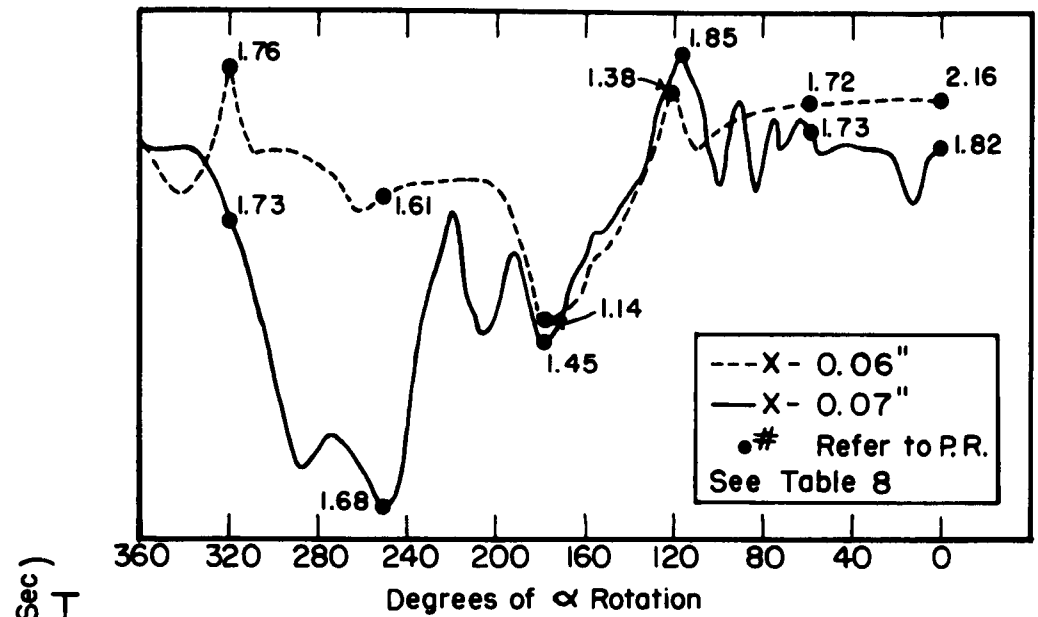
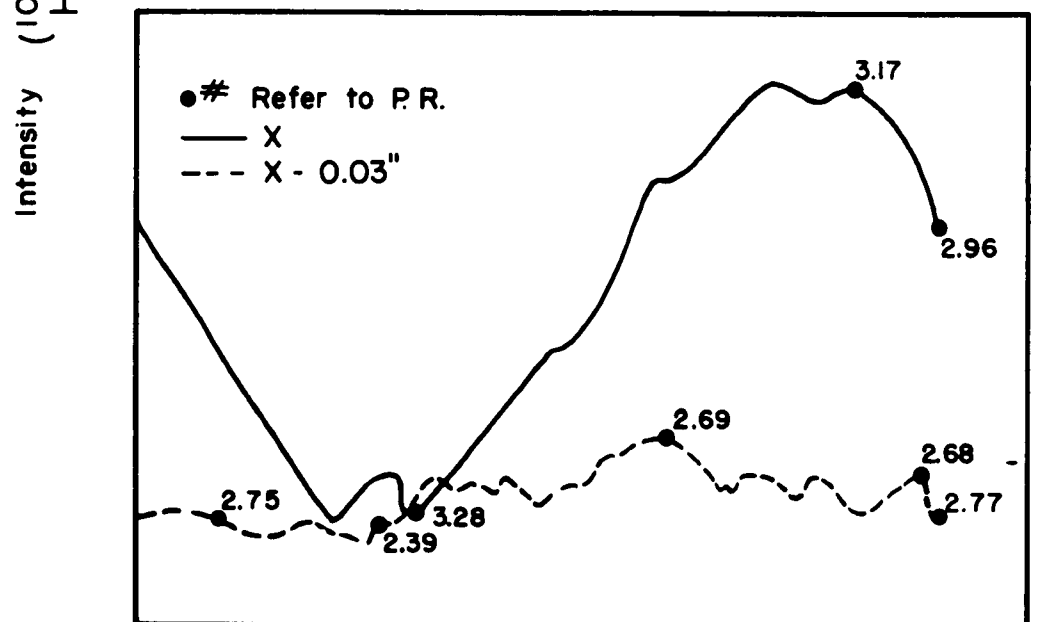


FIGURE 6 COMPARISON OF ϕ ROTATION FOR IDEAL ORIENTATION AND IDEAL RANDOM



a. Section Parallel to Seepage



b. Section Normal to Seepage

FIGURE 7

α ROTATION ON 002 PEAK FOR ISOTROPICALLY CONSOLIDATED KAOLINITE

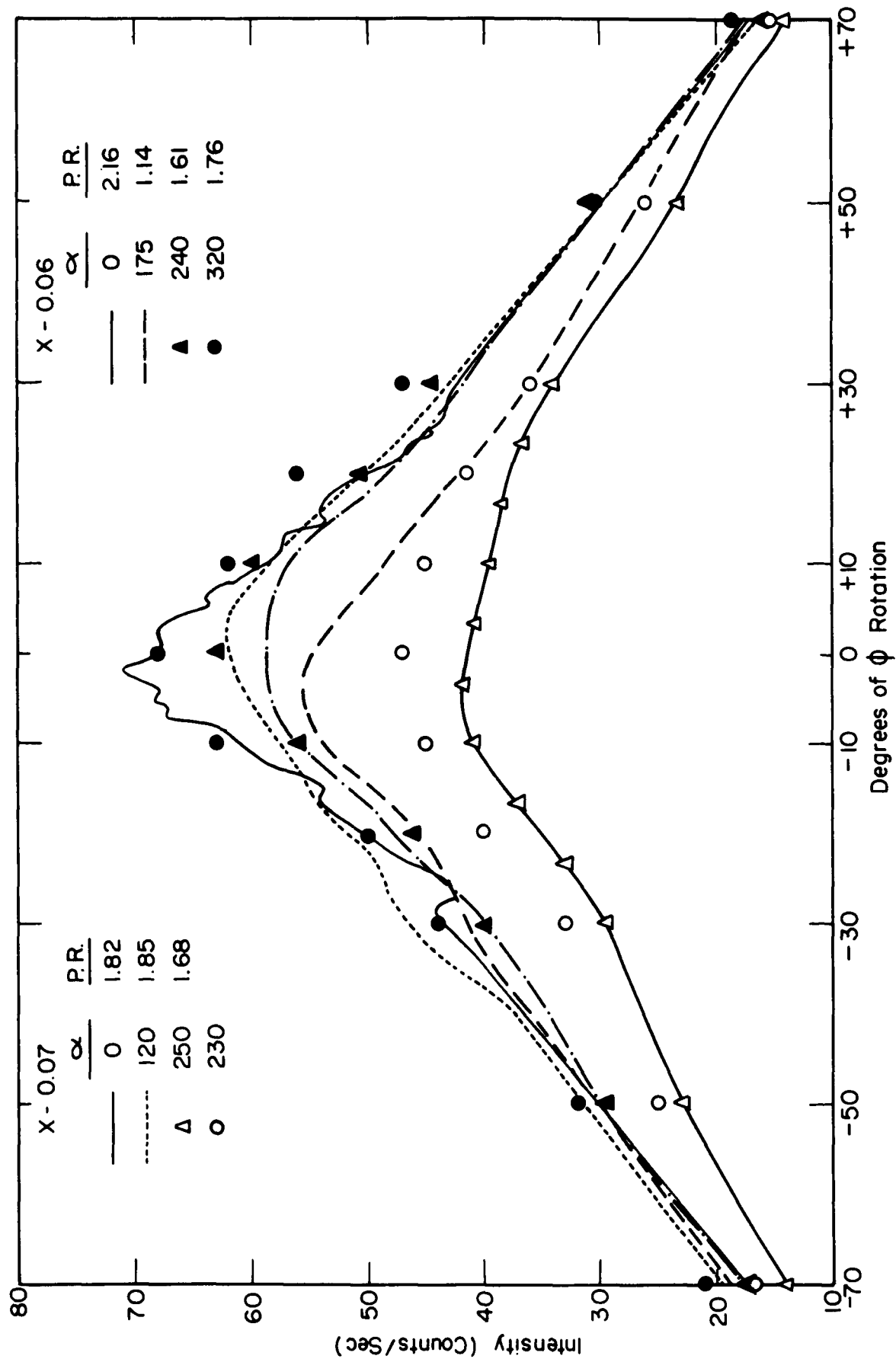


FIGURE 8 ϕ ROTATION ON 002 PEAK FOR ISOTROPICALLY CONSOLIDATED KAOLINITE PARALLEL TO SEEPAGE

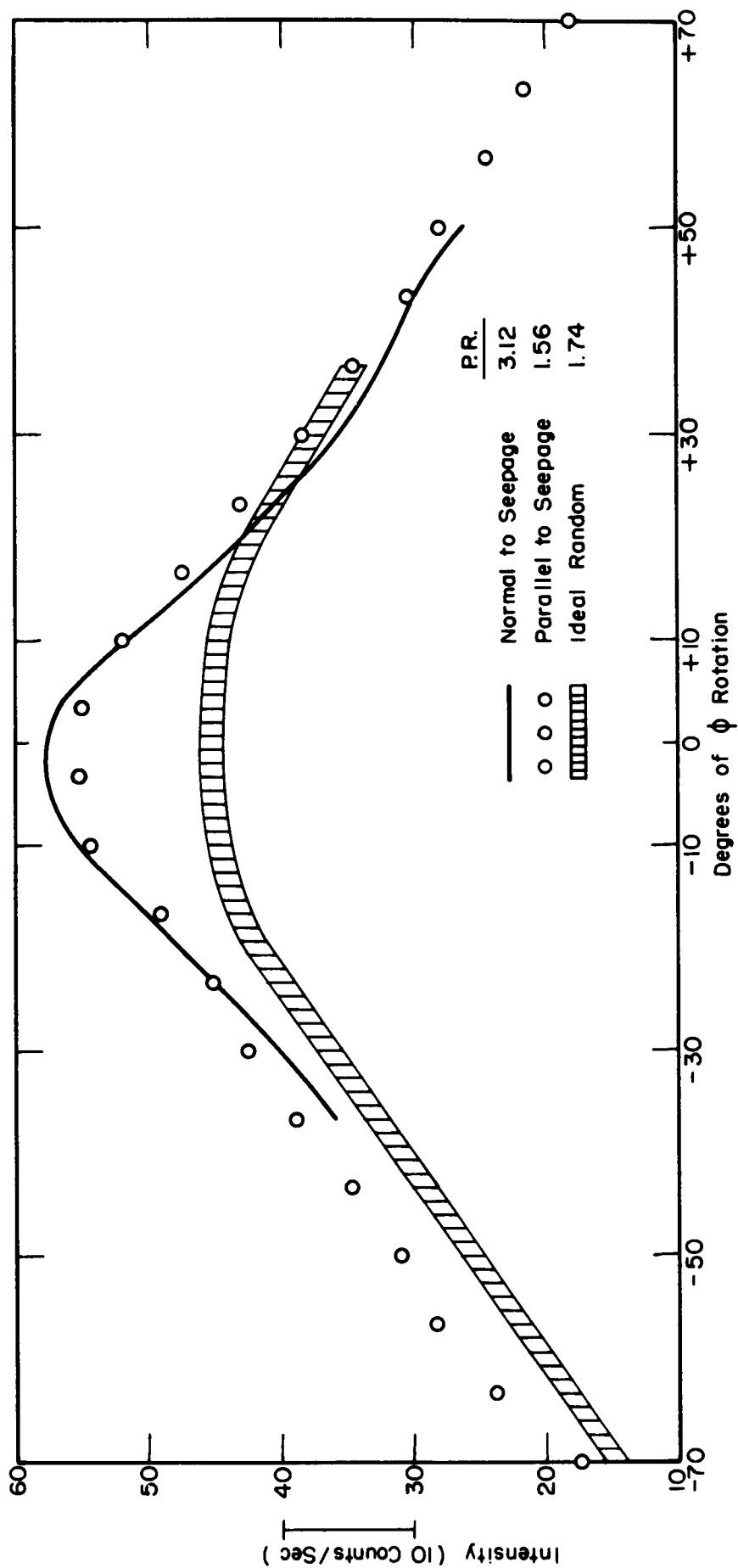


FIGURE 9 COMPARISON OF ϕ ROTATION FOR ISOTROPICALLY CONSOLIDATED KAOLINITE AND IDEAL RANDOM

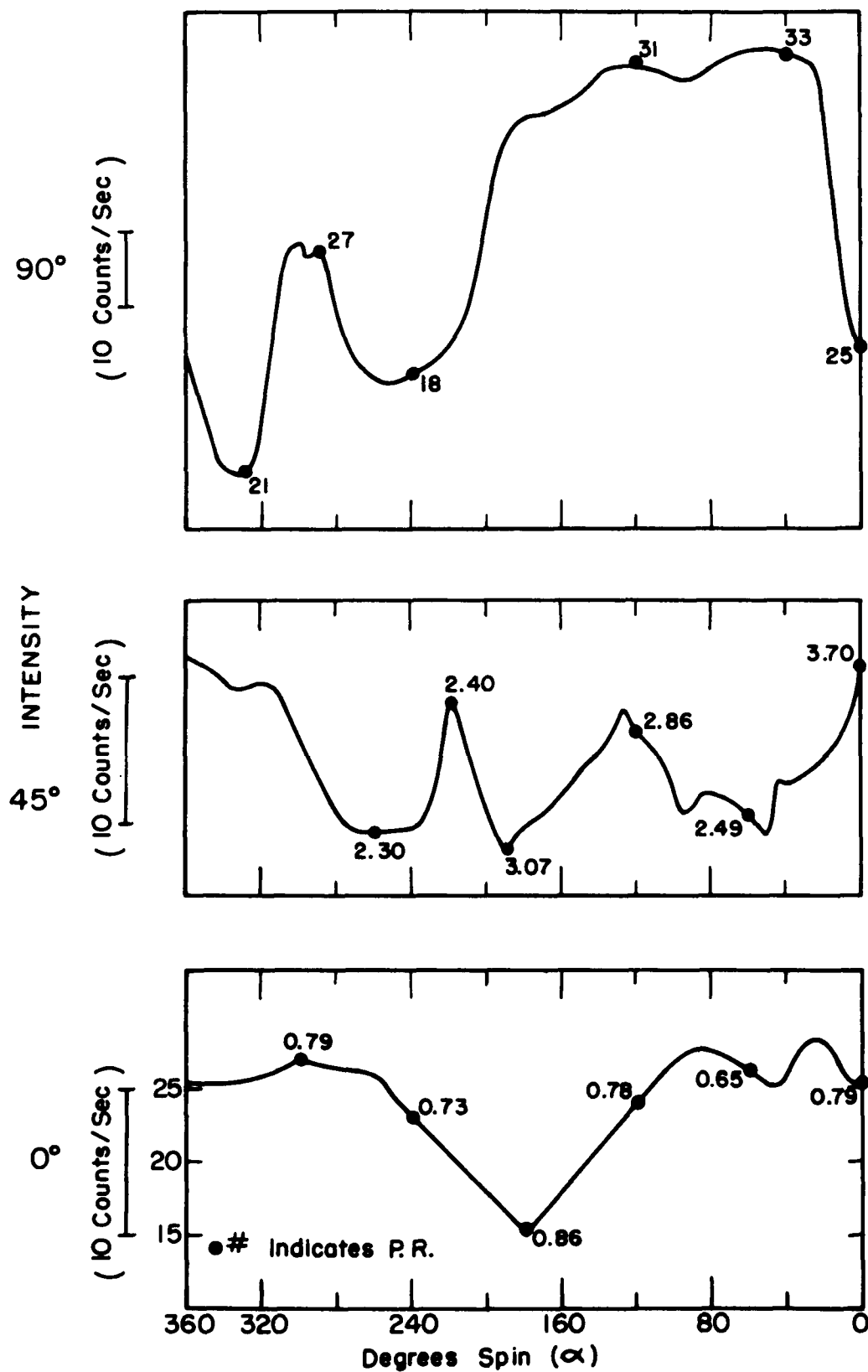


FIGURE 10 α ROTATION ON 002 PEAK FOR ANISOTROPICALLY CONSOLIDATED KAOLINITE (AT 0, 45 and 90° to $\bar{O}c$)

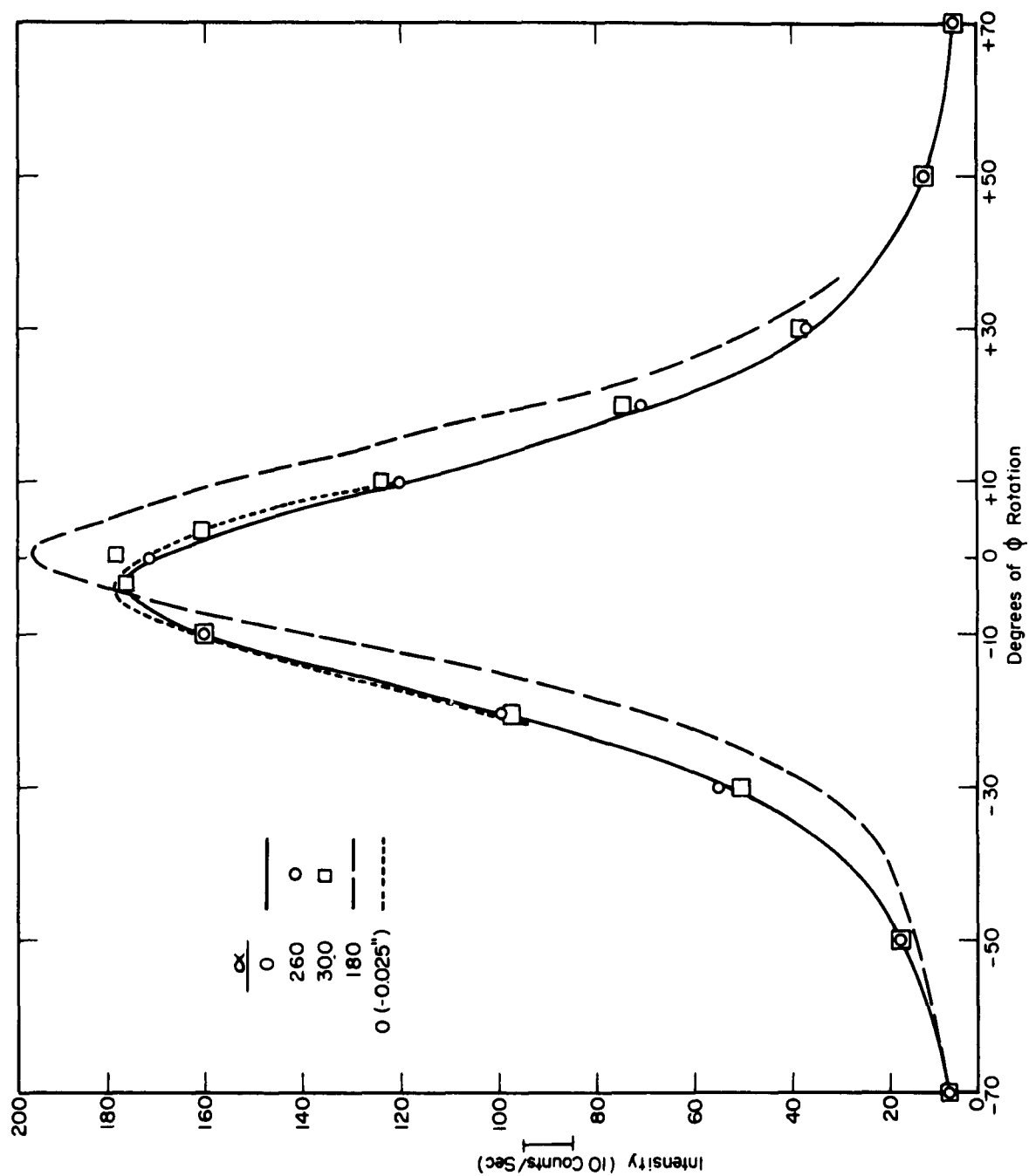


FIGURE II ϕ ROTATION ON 002 PEAK FOR ANISOTROPICALLY CONSOLIDATED KAOLINITE AT 90° TO $\bar{O}c$

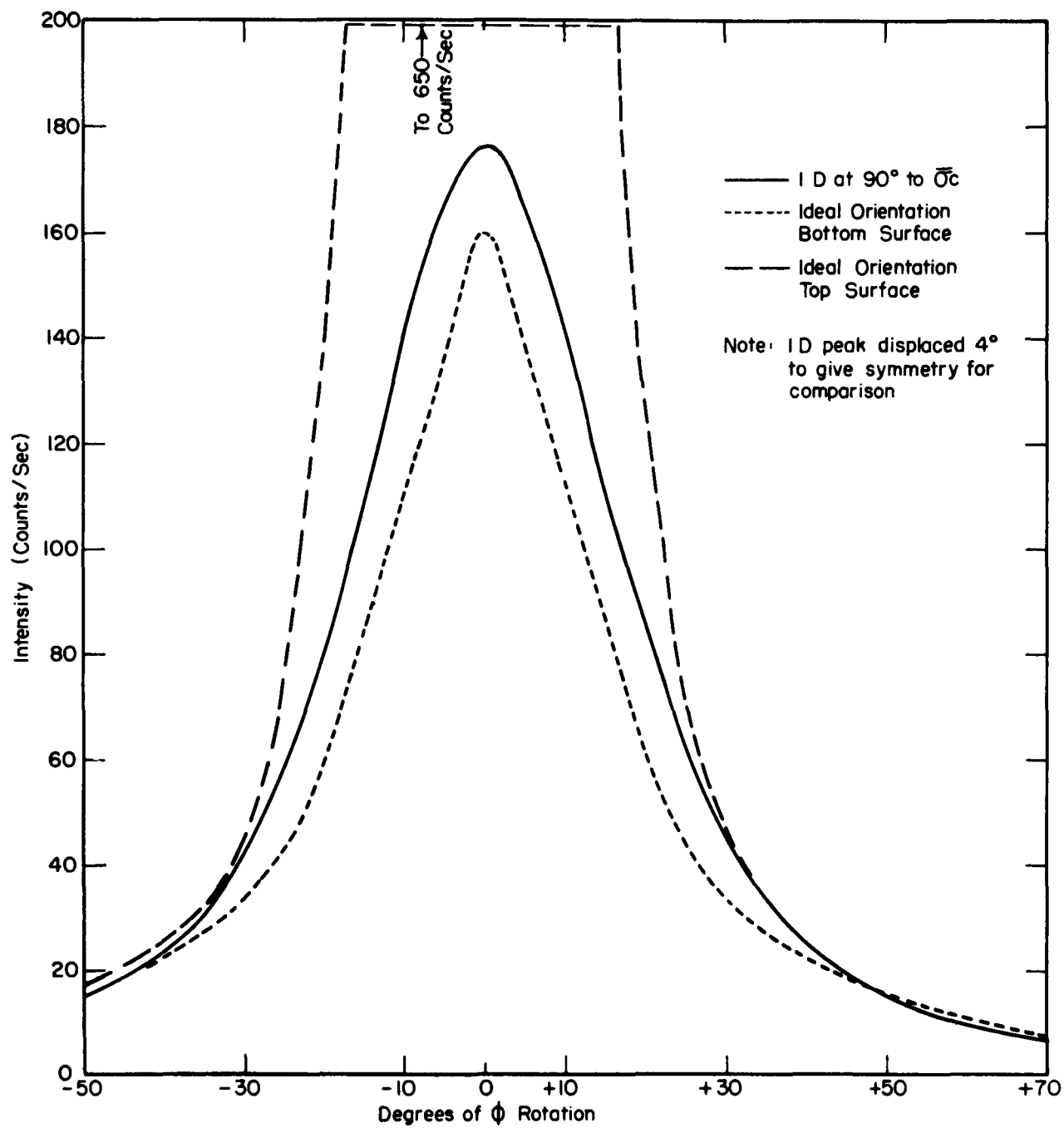


FIGURE 12 COMPARISON OF ANISOTROPICALLY CONSOLIDATED KAOLINITE AT 90° TO $\bar{O}c$ AND IDEAL ORIENTATION

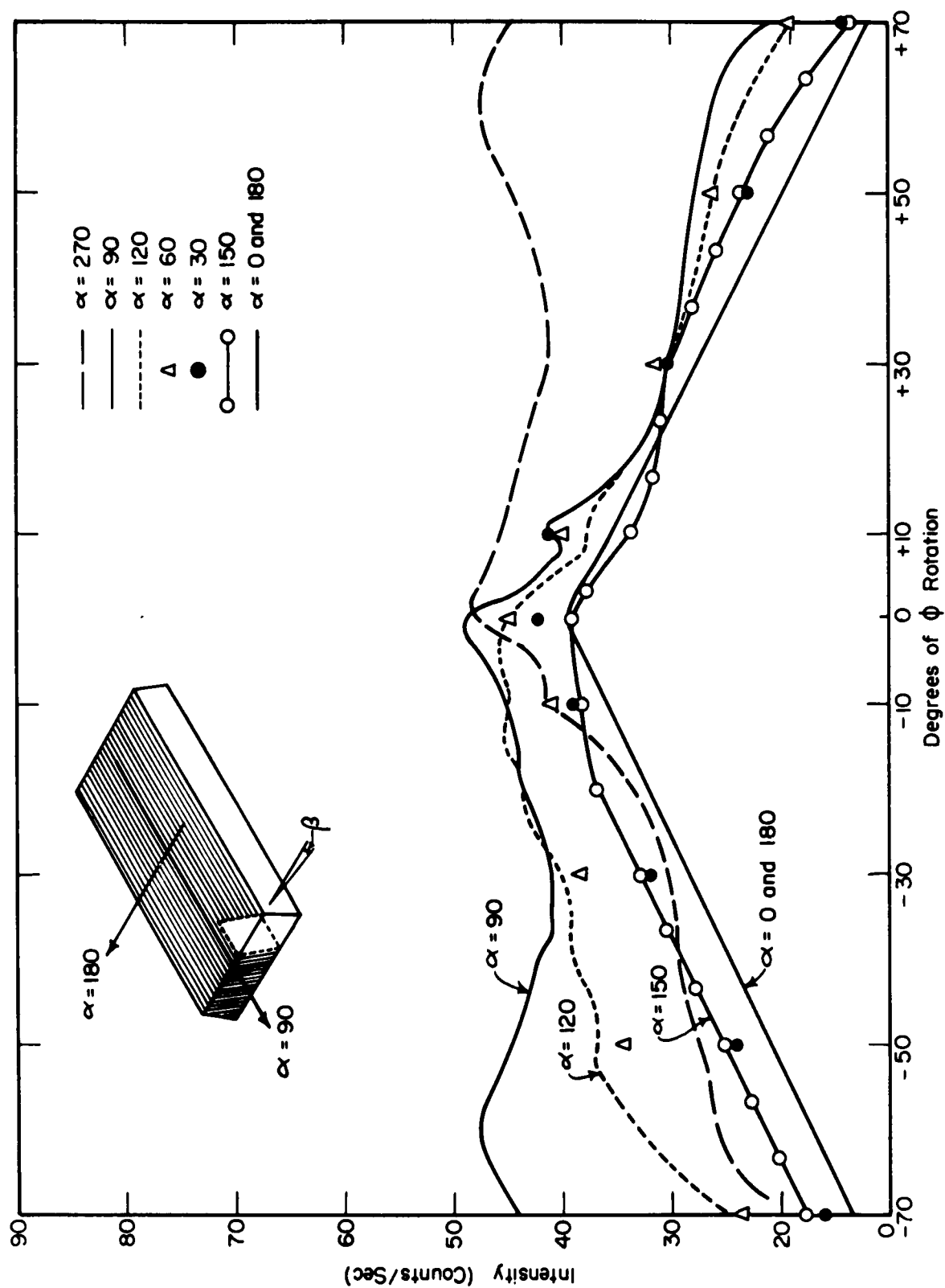


FIGURE 13 ϕ ROTATION ON 002 PEAK FOR ANISOTROPICALLY CONSOLIDATED KAOLINITE AT 0° TO 180° (VARIOUS α POSITIONS)

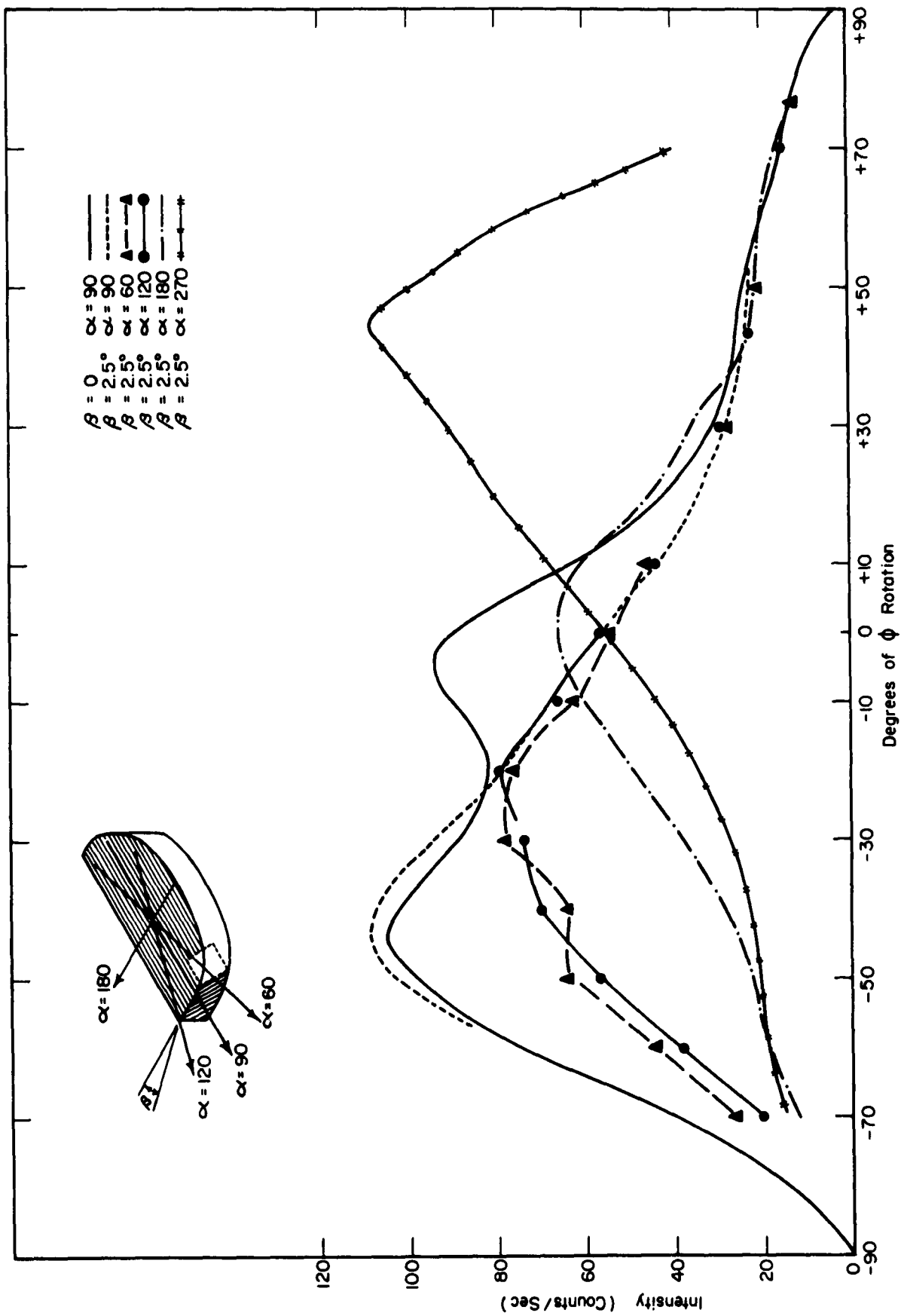


FIGURE 14 ϕ ROTATION ON 002 PEAK FOR ANISOTROPICALLY CONSOLIDATED KAOLINITE AT 45° TO σ_c

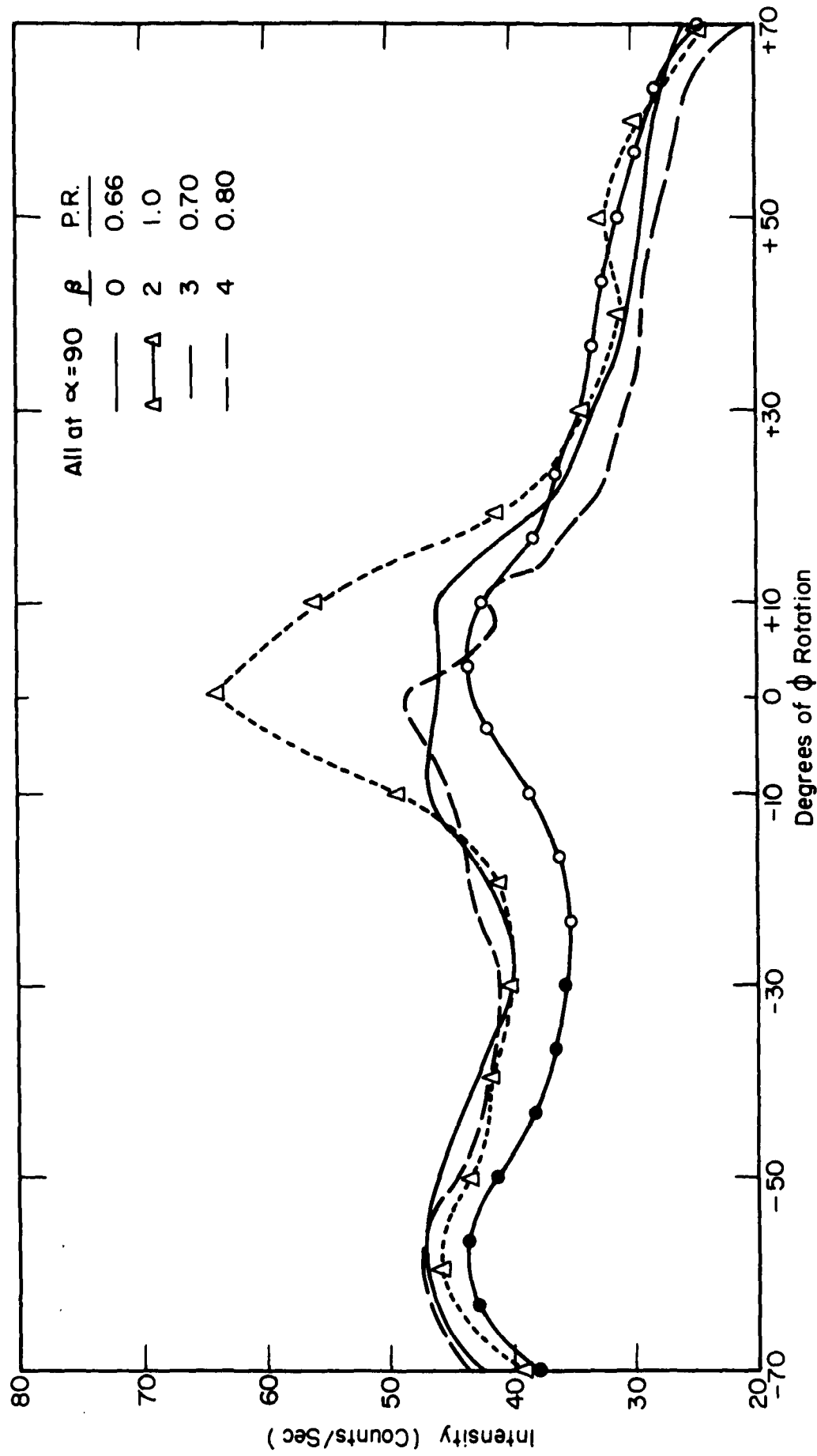


FIGURE 15 ϕ ROTATION ON 002 PEAK FOR ANISOTROPICALLY CONSOLIDATED KAOLINITE AT 0° TO 0°_c AS SURFACE TIPPED BY GRINDING

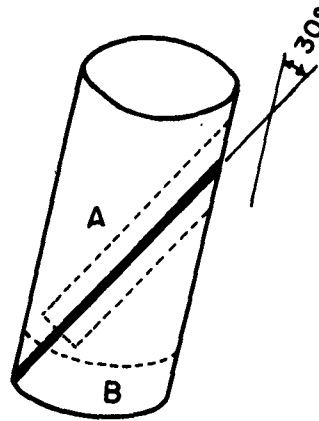
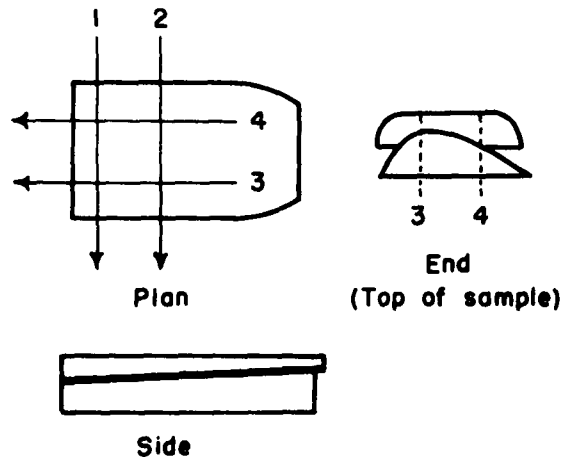


Diagram showing unconfined shear sample and specimen taken for x-ray examination.

Heavy black line is the visible shear line. Dotted lines indicate where sections A and B were taken from the sample.

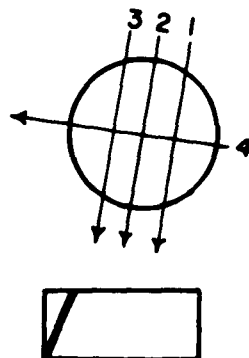
SECTION A



← Indicate direction and position of x-ray beam on the 4 spots studied in successive slices

Distance between spots 1 and 2, and spots 3 and 4 is 8.7 mm

SECTION B



Distance between spots 1 and 2 is 4 mm. Distance between spots 2 and 3 is 5 mm.

FIGURE 16 DIAGRAM SHOWING RELATION OF X-RAY SPECIMENS TO THE ORIGINAL UNCONFINED SHEAR SAMPLE

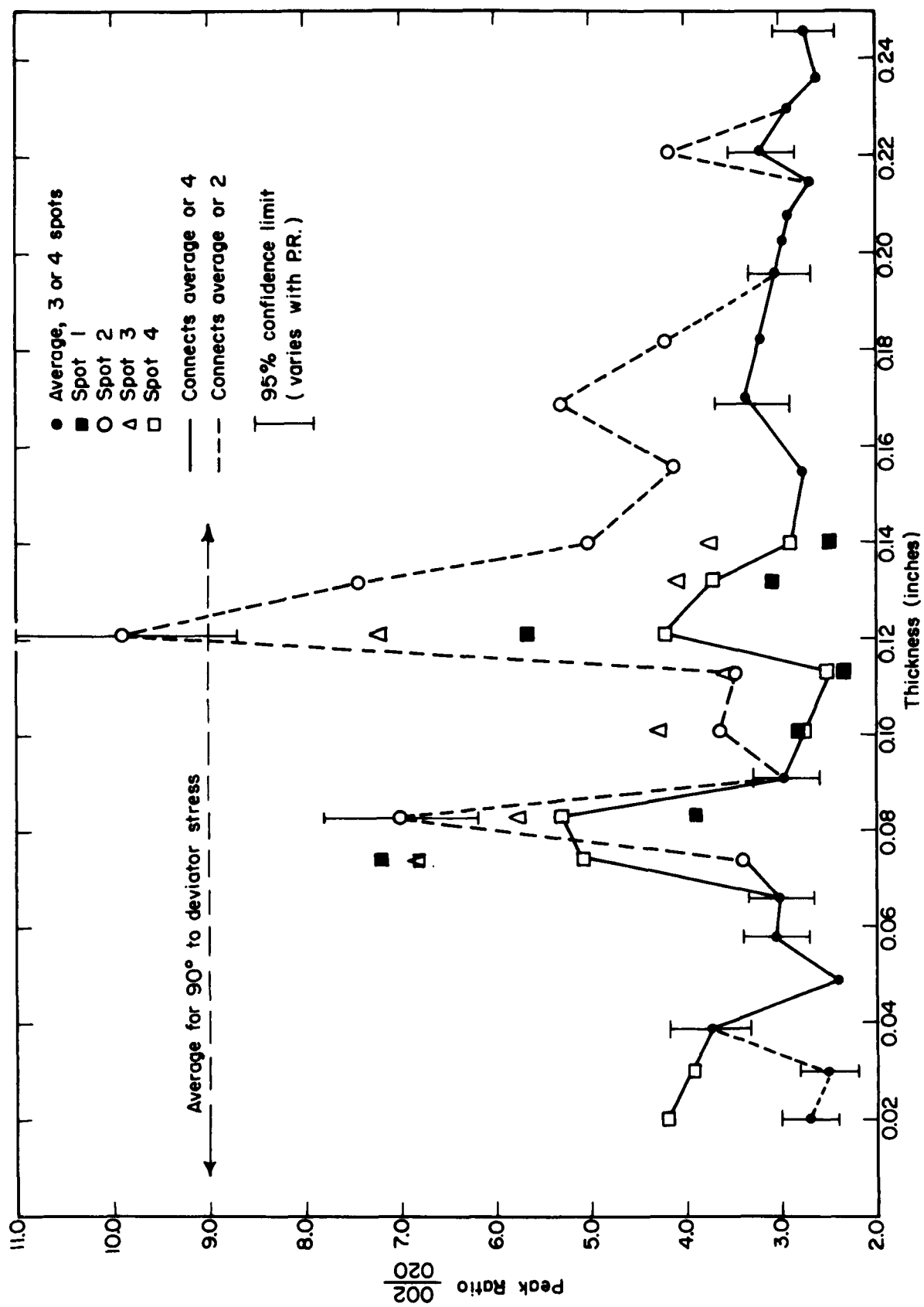


FIGURE 17 PARTICLE ORIENTATION ON SUCCESSIVE SLICES THROUGH A SHEAR ZONE

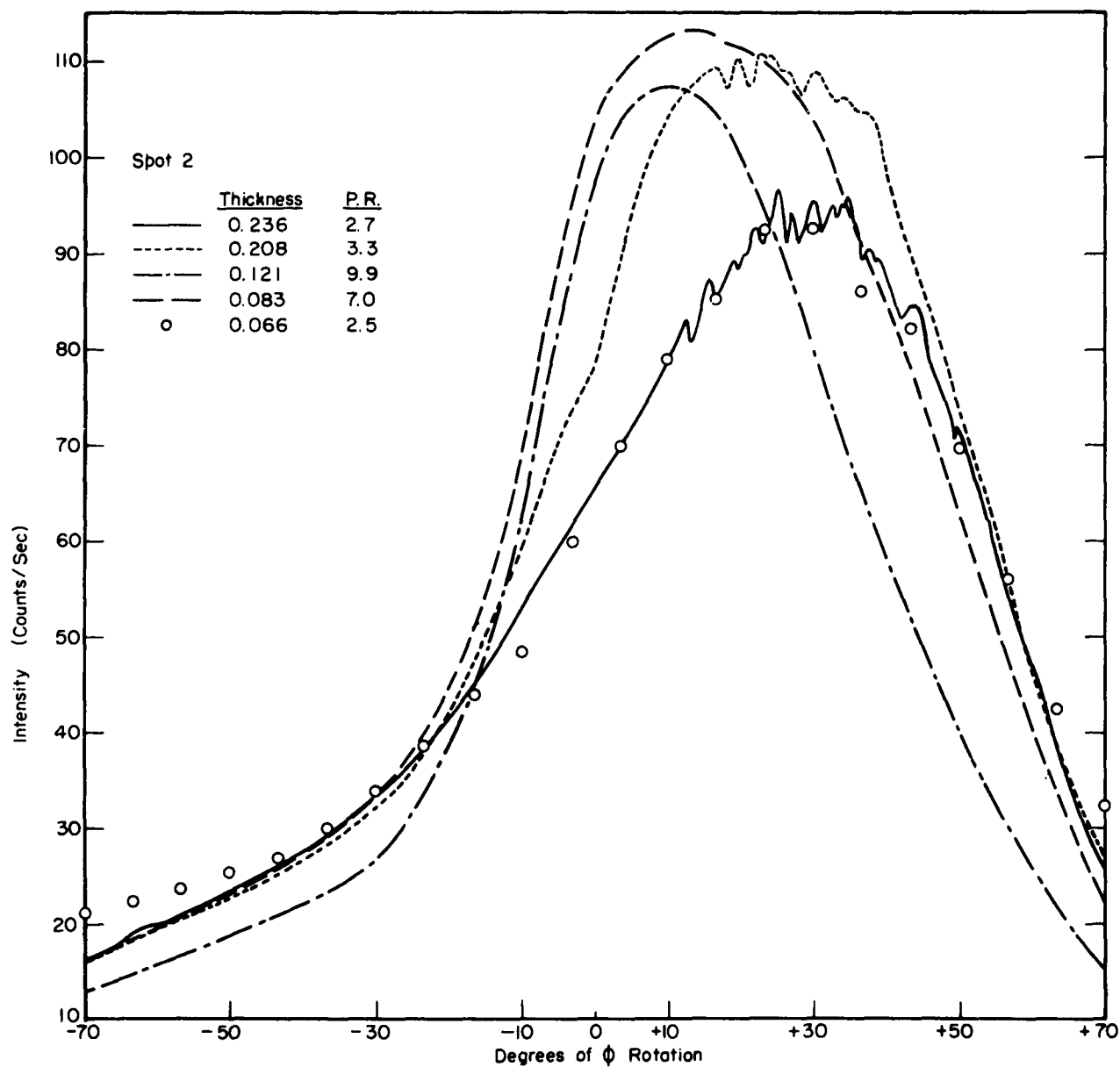


FIGURE 18 ϕ ROTATION ON 002 PEAK THROUGH THE SHEAR ZONE OF KAOLINITE
SUBJECTED TO UNCONFINED SHEAR: SPOT 2

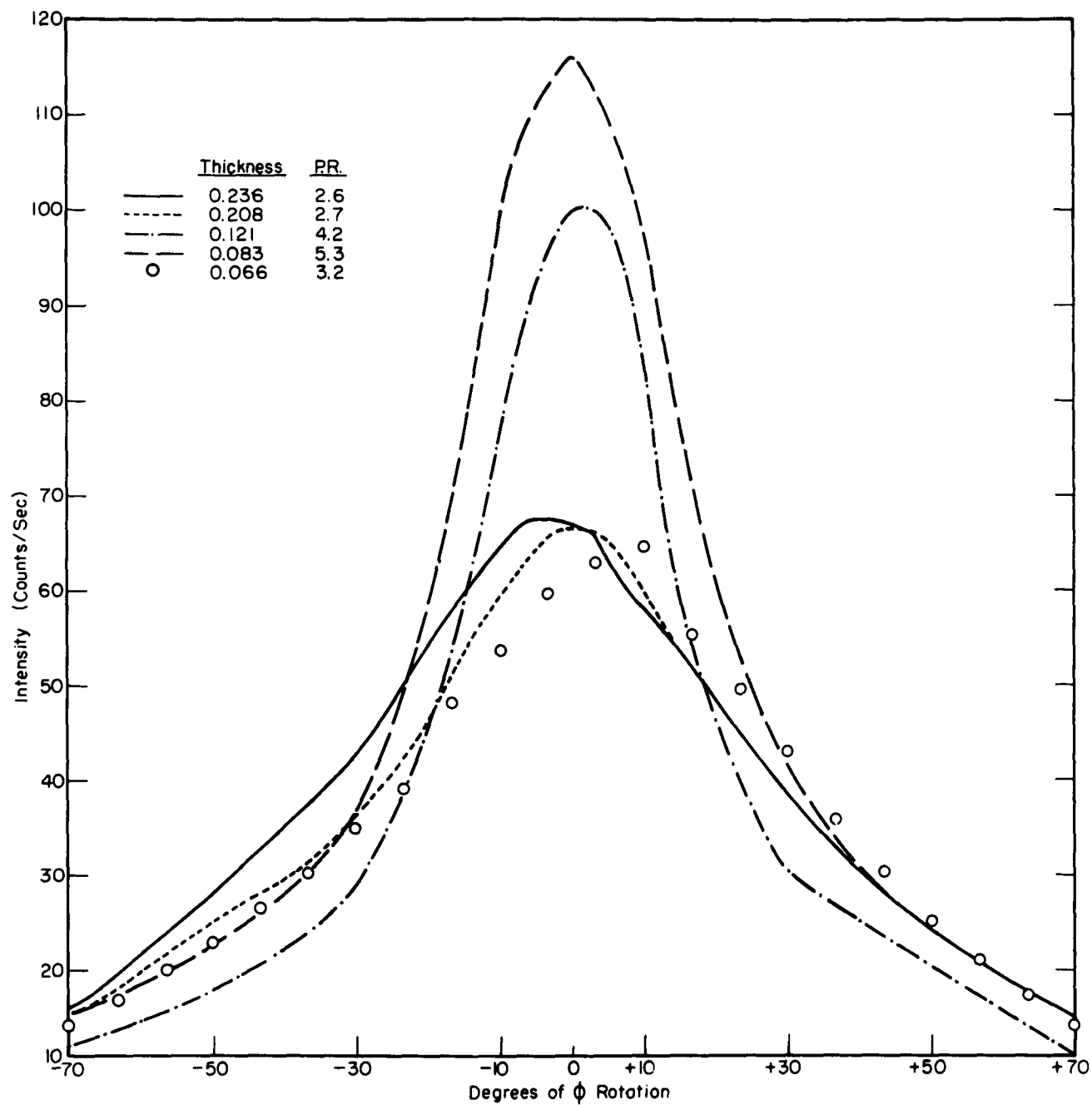


FIGURE 19 ϕ ROTATION ON 002 PEAK THROUGH THE SHEAR ZONE OF KAOLINITE SUBJECTED TO UNCONFINED SHEAR: SPOT 4

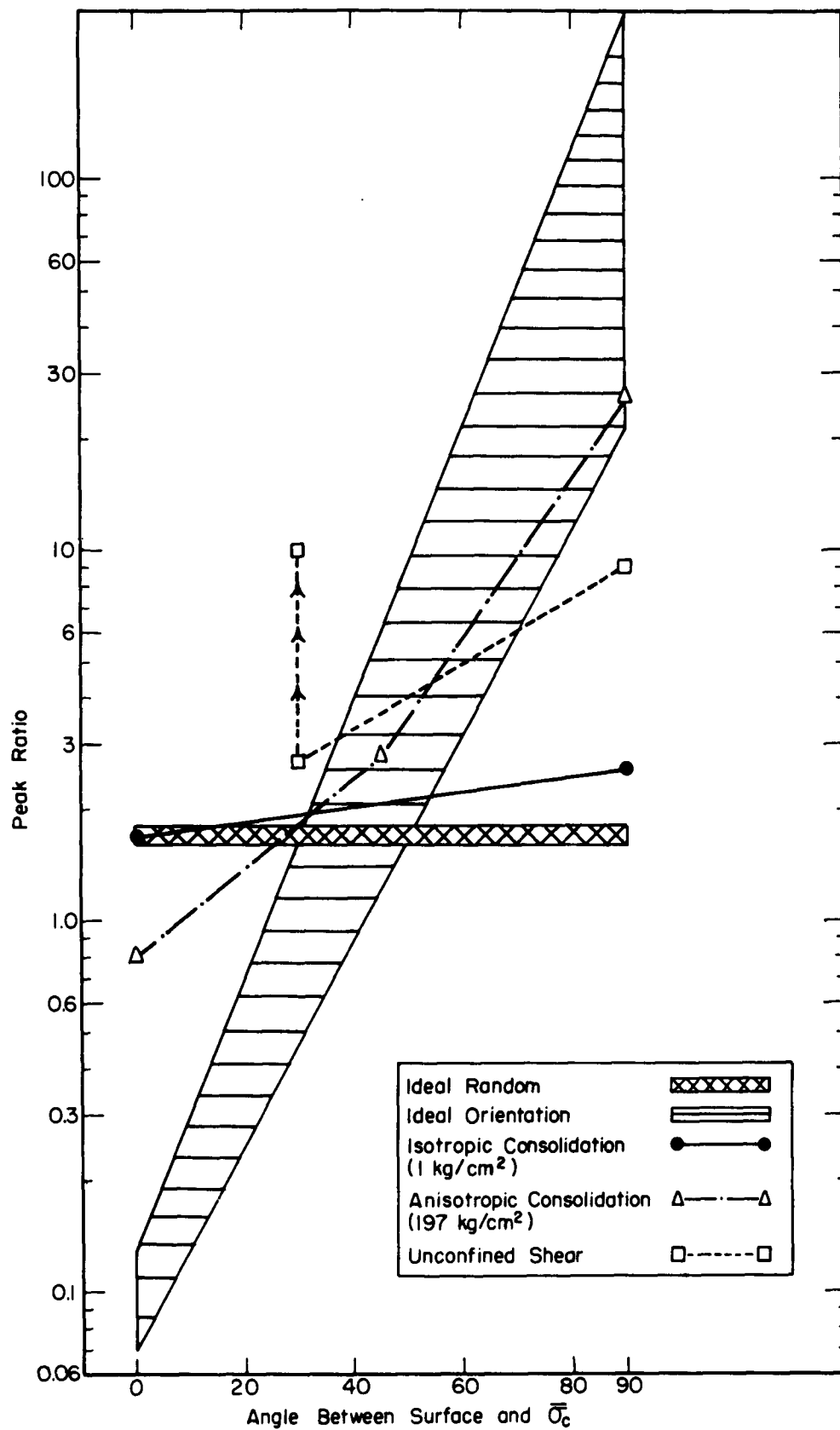


FIGURE 20 RELATION BETWEEN PEAK RATIO AND THE ANGLE BETWEEN THE SURFACE AND THE CONSOLIDATION PRESSURE FOR DIFFERENT KAOLINITES